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ANALYTICAL ABSTRACTS

1.—GENERAL ANALYTICAL CHEMISTRY

766. **Tin as an analytical reducing agent in glacial acetic acid.** N. D. Shah (Univ. of Wisconsin, Madison, U.S.A.). *Dissert. Abstr.*, 1957, **17**, 1316.—An investigation was made of the behaviour of metallic Sn in glacial acetic acid in the presence and absence of air, of its powers of reduction, and their application in the quant. determination of nitro, nitroso, org. nitrite, nitrate, and aldehyde groups. The reagent was found to be effective for nitro, nitroso and azo compounds, but not for nitrates or aldehydes. The presence of air did not cause interference with the determinations, and the method proved to be accurate to ± 1%. S.C.I. ABSTR.

767. **Syntheses of derivatives of dithizone and their application to analytical chemistry. I. *pp'*-Dimethyldithizone.** S. Takei, K. Shibuya and T. Kato. *Technol. Rep. Tôhoku Univ.*, 1957, **21** (2), 123–134.—Di-*p*-tolylthiocarbazone (*pp'*-dimethyldithizone) is a greenish-black powder decomposing at 153° to 156°. From spectrophotometric studies of CCl_4 soln. of this compound and its complexes with Zn, Cd, Pb and Hg, it is found that the introduction of the *p*-methyl groups results in an increase in the sensitivity of the reagent but no increase in selectivity. The derivative is a weaker acid than dithizone, and its Zn, Cd and Pb complexes are less stable than those of the unsubstituted dithizone. (See also Takei and Shibuya, *Anal. Abstr.*, 1957, **4**, 3215.)

II. *oo'*-Dimethyldithizone and its application to the determination of mercury and copper. S. Takei and T. Kato. *Ibid.*, 1957, **21** (2), 135–149.—*oo'*-Dimethyldithizone is a greenish-black powder, m.p. 164° (decomp.) after recrystallisation from CH_3OH - $CHCl_3$. This derivative is less reactive than dithizone itself but has an increased selectivity for Ag, Hg and Cu; practical procedures for the determination of Hg and Cu have been devised. For the determination of Hg, the pH of the sample soln. is adjusted to 1 to 3 (or > 3·5 if large amounts of Cl^- are present); Cu or large amounts of other metal ions can be screened by addition of EDTA. The soln. is shaken for 1 to 2 min. with 10 ml of *oo'*-dimethyldithizone in CCl_4 (50 mg per litre). The organic layer is passed through a dry filter-paper and its extinction measured at 486 mμ in a 10-mm cell. From 0 to 30 μg of Hg per 10 ml of CCl_4 can be determined with an error of about ±1%. For the determination of Cu, the pH of the soln. is adjusted to about 3 with Na acetate - $HClO_4$ buffer soln. and the soln. is then shaken for 5 min. with 10 ml of a CCl_4 soln. of *oo'*-dimethyldithizone (50 mg per litre). The organic layer is passed through a dry filter-paper to remove emulsified water and its extinction is measured at 538 mμ in a 10-mm cell. From 0 to 10 μg of Cu in 10 ml of CCl_4 can be determined with an error of about ±1%, but Hg interferes. A. JOBLING

768. **Simplified procedure for preparation of Girard's reagent T.** R. N. Chakravarti and M. N. Mitra (Dept. Chem., School Tropical Medicine, Calcutta). *J. Instn Chem. India*, 1957, **29** (3), 158–160.—In the modified procedure described, ethyl chloroacetate (**I**), precooled to below 0°, is added to an alcoholic soln. of trimethylamine cooled to about − 10°. No stirrer or spiral condenser is used, but a gentle swirling is helpful during additions of **I** and hydrazine hydrate. O. M. WHITTON

769. **Preparation of optically pure light petroleum.** B. van der Ven and A. P. de Jonge (Unilever Res. Lab., Vlaardingen, The Netherlands). *Rec. Trav. Chim. Pays-Bas*, 1957, **76** (2), 169–172 (in English).—A procedure for the preparation of light petroleum which is transparent down to about 220 mμ is described. The light petroleum is freed from aromatic hydrocarbons by nitration with a mixture of equal vol. of red fuming HNO_3 and conc. H_2SO_4, washing in succession with water, 20% KOH soln., and water, followed by fractionation over a 1-m column of Raschig rings and chromatography through a column of activated carbon. The product can then be re-fractionated through a Vigreux fractionating column and finally passed down a column of alumina to remove any remaining traces of nitro compounds. P. S. STROSS

770. **Diphenylamine as an internal indicator for diazotisation titrations.** Tao Duenn (First National Pharm. Plant, Shanghai). *Acta Pharm. Sinica*, 1957, **5** (2), 97–105.—For the determination of aromatic amines, sulpha drugs, local anaesthetics, etc., by titration with NO_2^-, diphenylamine is an excellent indicator. N. E.

771. **Metallochromic indicators. III. Preparation of 3:3'-bis-NN-di(carboxymethyl)aminomethylthymolsulphonephthalein (methylthymol blue).** J. Körbl (Res. Inst. Pharm. and Biochem., Prague, Czechoslovakia). *Chem. Listy*, 1957, **51** (7), 1304–1306.—The preparation of a new metallochromic indicator for complexometric titrations, methylthymol blue (Körbl and Přibil, *Anal. Abstr.*, 1958, **5**, 10), in the form of a crystalline, chromatographically pure sodium salt is described.

IV. Note on the preparation and properties of calcein. J. Körbl and F. Vydra. *Ibid.*, 1957, **51** (8), 1457–1461.—The complexometric indicator calcein was prepared by a modified procedure. In contradiction to the statements of Diehl and Ellingboe (*Anal. Abstr.*, 1956, **3**, 2653), it has been found that this indicator does not yield colour changes, but very distinct fluorescence changes that can be used for accurate complexometric determinations of Ca, Sr and Ba in coloured soln. This indicator can also be used for very sensitive fluorimetric determinations of certain metals on a micro scale. J. ZÝKA

772. **Accurate measurement of pH. II. Sources of experimental error in pH measurement.** G. Mattock (Electronic Instruments Ltd., Richmond, Surrey, England). *Lab. Practice*, 1957, **6** (9), 521–525.—The sources of error in pH measurement, arising from junction potentials, salt effects, and from the reference and glass electrodes, are reviewed. Details are given of the different effects that influence the junction potential, regarded as the principal source of error. These errors assume particular importance if accuracies $> 0{\cdot}02$ of a pH unit are required. J. H. WATON

773. **Titrations in non-aqueous solvents. I.** R. van Poucke (N.V. Gevaert-Photoproducten, Mortsel, Antwerp). *Ind. Chim. Belge*, 1957, **22** (3), 271–283.—A review is presented of the literature on the basic theory of titration in non-aq. solvents and of the titration in non-aq. media of carboxylic acids, acid chlorides and anhydrides, thiols, sulphonamides, enols and imides, phenols, amino acids, oximes and pyrroles. (48 references.) S.C.I. ABSTR.

774. **Paper chromatography and paper electrophoresis. [II.]** A. Grüne (C. Schleicher and Schüll, Dassel, W. Germany). *Chimia*, 1957, **11** (8), 213–256.—A review is presented of the use of these techniques in the determination of structure or identification of proteins, antibiotics, vitamins, alkaloids, sugars, aliphatic acids and related compounds, amines, phenols, aromatic hydroxy and amino acids, phosphate in both organic (*e.g.*, nucleic acids) and inorganic compounds, dyestuffs and cations. E. G. CUMMINS

775. **Colorimetric analysis with organic reagents.** J. H. Yoe (Univ. of Virginia, Charlottesville, U.S.A.). *Anal. Chem.*, 1957, **29** (9), 1246–1251.—The development of colorimetric analysis is discussed, special reference being made to the importance and measurement of pH, instrumental techniques, and the use of ion-exchange resins and water-immiscible organic liquids. Several organic colorimetric reagents are discussed. G. P. COOK

776. **Mathematics of colorimetric analysis with the ring oven.** W. Knödel and H. Weisz (Tech. Hochschule, Vienna, Austria). *Mikrochim. Acta*, 1957, (3–4), 417–420 (in German).—The semi-quant. analysis with a ring oven previously described (*Mikrochim. Acta*, 1954, 140, 376; 1956, 667), with only visual estimation, can lead to useful results, since a weighted mean is taken. For amounts of material of $\simeq 2$ μg, the error is $\simeq 5\%$. J. H. WATON

777. **Measurements of dielectric constants in analytical chemistry.** S. Wolf (Metrohm. A.-G., Herisau, Switz.). *Chemikerztg*, 1956, **80**, 552–557.—A review with 34 references.

2.—INORGANIC ANALYSIS

778. **Gas-chromatographic separation of some permanent gases on silica gel at reduced temperatures.** D. E. Szulczewski and T. Higuchi (School of Pharm., Univ. of Wisconsin, Madison, U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1541–1543.—Mixtures of gases such as oxygen, nitrogen, nitric oxide and carbon monoxide can be separated at the temp. of solid CO_2 - acetone on a 6-ft. silica gel column. The method has been applied to gases present in ampoules of old amyl nitrite. K. A. PROCTOR

779. **Separation of group IIb and IIIa metals by paper chromatography.** T. Barnabas and J. Barnabas (Ahmednagar Coll., Ahmednagar, India). *Naturwissenschaften*, 1957, **44** (3), 61–62 (in English).—The solvent system glacial acetic acid - acetone - water (8:1·5:2·5) was found to give a good separation of the chlorides of As, Sb and Sn. The approx. R_F values on circular paper chromatograms were As (0·59), Sb (0·76) and Sn (0·92). The best solvent system for the chlorides of Fe, Al and Cr was glacial acetic acid containing 20% (v/v) of 5 *N* HCl. N. E.

780. **Chromatographic determination of micro amounts of metals.** A. Lacourt and P. Heyndryckx. *Chem. Age*, 1957, **78**, 251.—Microgram quantities of Co, Cu, Zn, Cd, Pb and Ba may be determined on paper chromatograms, with 8-hydroxyquinoline in acetone - acetic acid as spraying agent, with the procedure previously described (*Mikrochim. Acta*, 1955, (1), 61; 1956, (9), 1389; (11), 1621, 1685). The extinction of the spot at 360 mμ is determined 30 min. after spraying, by placing the coloured area in water in a cuvette, with a sprayed paper blank as reference. The limits of detection are 0·03 μg for Co and Cu, and 0·02 μg for Zn. D. G. LLOYD

781. **Use of anion exchangers in analytical chemistry. I. Sorption of the chloride complexes of certain metals on the anion-exchange resin OAL.** K. Liška and L. Klír (Res. Inst. Metals, Panenské Břežany, Czechoslovakia). *Chem. Listy*, 1957, **51** (8), 1467–1469.—The elution constants of the chloride complexes of Ni, Mo, As, Co, Cu, Zn, Fe, Pb, Cd, Bi and Sn on the strongly basic anion-exchange resin OAL were measured and the possibilities of analytical separation of these elements were studied. J. ZÝKA

782. **Use of anion exchangers in analytical chemistry. III. Separation of small amounts of lead, zinc, cadmium, tin and bismuth from copper, cobalt and iron.** K. Liška and L. Klír (Res. Inst. Metals, Panenské Břežany, Czechoslovakia). *Chem. Listy*, 1957, **51** (8), 1549–1550.—The elution constants of these elements on a strongly basic anion exchanger OAL were measured and their dependence on the concn. of HCl used for various analytical separations is shown. With the use of this method, the impurities in pure copper (*e.g.*, 0·0008% of Cd), cobalt or iron can be determined. J. ZÝKA

783. **Chemical indicators. IV. Complexes of catechol violet with ter- and quadri-valent metals.** O. Ryba, J. Cífka, D. Ježková, M. Malát and V. Suk (Inst. Anal. Chem., Charles' Univ., Prague, Czechoslovakia). *Chem. Listy*, 1957, **51** (8), 1462–1466.—The complexes of catechol violet (**I**) with Bi, Zr, Sn, Ti, Th, Ga, Al, In, Sb and BO_3^{3-} were studied by the use of colorimetric methods. The stability constants of **I** with Bi, Zr, Th, Ga, Al and In are discussed. J. ZÝKA

784. **Reaction rates in the analytical determination of some inorganic peroxides and superoxides.** S. H. Cohen and J. L. Margrave (Dept. of Chem., Univ. of Wisconsin, Madison, U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1462–1463.—The rate of evolution of oxygen when peroxides and superoxides are decomposed in water is determined for the compounds

of the alkali and alkaline-earth metals. It is demonstrated that, whilst oxygen evolution is slower from peroxides than from superoxides, the different rates do not enable a clear-cut analytical separation to be made. From the results it is established that commercial (yellow) sodium peroxide contains, at most, 6% of sodium superoxide. D. A. PANTONY

785. **The spectrochemical determination of lithium in Cornish china clay.** E. H. Henderson, M. J. Owers and M. S. W. Webb [U.K.A.E.A. (Res. Group), Woolwich Outstation]. A.E.R.E. Report C/R 2137, 1957, 13 pp.—The lithium content of finely ground micaceous and siliceous materials may be determined by either emission spectroscopy or flame photometry over the range 0·05 to 0·4% to within ±5%. *Procedure*—Three dilutions are prepared with $BaCl_2$ containing 3·5% of sodium silicate, and the Seidel densities of the lines Li 6103·7, Na^{I} 5670·2 and Na^{II} 5675·7 v are determined; from the functions $S_{Na} - S_{Na^{I}}$ and $S_{Li} - S_{Na^{II}}$ the content of Li may be obtained after calibration with Li_2CO_3 as primary standard. The sensitivity is 0·01%. No interference by other anions or cations was found. D. G. LLOYD

786. **X-ray spectrographic determination of caesium and rubidium.** J. M. Axelrod and I. Adler (U.S. Geograph. Survey, Washington, D.C., U.S.A.). *Anal. Chem.*, 1957, **29** (9), 1280–1281.—Caesium and Rb can be determined by X-ray spectroscopy in mica, beryl and felspar with a precision within 10% of the amount present. The sensitivity is 0·1% for Cs and 0·02% for Rb, and the results agree well with those obtained by flame photometry and radioactivation. K. A. PROCTOR

787. **New spectrophotometric method for determination of copper with sodium versenate [EDTA, disodium salt].** N. A. Ramaiah and Vishnu (Dept. Phys. Chem., Indian Inst. of Sugar Technol., Kanpur). *Proc. Indian Acad. Sci., A*, 1957, **45** (2), 113–116.—A new method is described for the determination of Cu^{2+} by EDTA (disodium salt), in which the absorption of the copper ammonium complex at 630 mμ is used. The experimental error is < 1%. I. JONES

788. **Determination of copper in nickel solutions.** T. A. Downey (Hanson-Van Winkle-Munning Co., Grand Rapide, Mich., U.S.A.). *Plating*, 1957, **44**, 383–385.—The colorimetric method is based on the interaction of Cu^{I} with neocuproine. As little as 1 mg of Cu per litre can be determined with an accuracy of better than 5%. None of the common impurities interfere. CHEM. ABSTR.

789. **Spectrophotometric determination of copper in [sea] salt.** J. M. Rafols Rovira ('Aprovechamientos Salineros, S.A.', Barcelona). *Inf. Quim. Anal.*, 1957, **11** (4), 99–102.—Trace amounts of Cu in sea salt are determined by reaction with Na diethyldithiocarbamate, extraction of the complex with xylene and immediate measurement of the extinction of the extract at 436 mμ. The content of Cu of samples from three sources taken over a period of four years was in the range 0 to 0·07 p.p.m. G. H. FOXLEY

790. **Determination of iron, nickel and lead in blister and refined copper.** S. Barabas and W. C. Cooper (Canadian Copper Refiners, Ltd., Montreal East, Quebec, Canada). *Metallurgia, Manchr.*, 1957, **56**, 101–103.—The sample is dissolved in HNO_3 and Cu is electro-deposited from the slightly acid solution. Iron and Ni are determined colorimetrically on small aliquots, with NH_4SCN and dimethylglyoxime, respectively, as reagents. Lead is estimated polarographically in HCl. Some typical results are given. S.C.I. ABSTR.

791. **Precipitation of magnesium with ethylenedinitrilotetra-acetic acid [EDTA].** C. E. Bricker and G. H. Parker (Frick Chem. Lab., Univ., Princeton, N.J., U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1470–1474.—The variables in pptn. of Mg^{2+} with EDTA (disodium salt) are investigated, and properties of the product are described. The product, of formula $MgC_{10}H_{14}O_8N_2.6H_2O$, can be used as a weighing form for Mg^{2+} after air-drying, or, as the anhydrous salt, after vacuum-drying. A pH of 3·5 to 4·0 is recommended for pptn., but the rate of deposition is very slow (48 hr. for 99·96% recovery). There is no interference from Ba^{2+}, Ca^{2+}, Li^{+}, Co^{2+}, Ni^{2+}, Zn^{2+}, Cd^{2+}, Th^{4+}, Bi^{3+}, acetate and borate except in high concn., but Be^{2+} impairs the filtering properties of the ppt. and Al^{3+}, Mn^{2+}, PO_4^{3-}, tartrate and citrate cause low results. D. A. PANTONY

792. **Determination of calcium as [luminescence] activator in alkali-halide crystals.** J. Trnka (Inst. Tech. Physics, Acad. Sci., Prague, Czechoslovakia). *Chem. Listy*, 1957, **51** (7), 1378.—The content of Ca (3×10^{-5} *M* to 5×10^{-3} *M*) in crystals of NaCl or KCl can be determined polarographically. Calcium reacts in aq. NH_3 medium with the zinc complex of EDTA (disodium salt), setting free an equiv. amount of Zn^{2+}, which yield a well-developed polarographic wave (*cf.* Přibil and Vicenová, *Chem. Listy*, 1952, **46**, 535). Sodium chloride from the soln. of the sample must be partially removed by pptn. with HCl and ethanol. J. ZÝKA

793. **Complexometric titrations (chelatometry). XXXI. Estimation of calcium ethylenediaminetetra-acetate.** F. Buben, J. Körbl and R. Přibil (Res. Inst. Pharm. and Biochem., Prague, Czechoslovakia). *Chem. Listy*, 1957, **51** (7), 1307–1311.—The complexometric determination of free Ca^{2+}, EDTA and total Ca is described. *Procedure*—Dissolve the sample (200 to 300 mg) in H_2O (100 ml), add methylthymol blue as indicator (0·1% soln.) (3 to 5 drops) and 0·1 *N* NaOH (5 ml); when free Ca^{2+} are present the yellow colour changes to blue. Titrate with 0·01 *M* EDTA (disodium salt) to a pale grey. The free EDTA in the sample can be titrated with 0·01 *M* $CaCl_2$ (blue colour). Then add *N* HNO_3 (0·8 ml) and hexamine (0·25 g). The yellow soln. can be titrated with 0·05 *M* $Pb(NO_3)_2$ until blue and thus the total amount of EDTA in the sample can be determined. Add 0·05 *M* EDTA (disodium salt) (1 drop) (colour changes to yellow) and aq. NH_3 (25%) (10 ml) (the blue colour of the complex of Ca with the indicator appears), and titrate the total Ca with 0·05 *M* EDTA (disodium salt) until slightly grey. The content of water in the sample can be determined with the use of the Fischer method or thermogravimetrically. The method is suitable for use in the analysis of some pharmaceutical preparations. J. ZÝKA

794. **Diethyldithiocarbamate as volumetric reagent. II. Volumetric determination of mercury.** V. Šedivec (Inst. Hyg. Ind. Diseases, Prague, Czechoslovakia). *Chem. Listy*, 1957, **51** (8), 1471–1474.—The addition of EDTA and pyridine prevents

the pptn. of diethyldithiocarbamates and of hydroxides of some metals when titrating Hg with diethyldithiocarbamate with a Cu^{2+} indicator. *Procedure*—To the sample containing 1 to 12 mg of Hg add 0·1 *N* EDTA (disodium salt) (1 to 5 ml) and adjust the vol. to 20 ml. Add the same vol. of pyridine and 1 drop of 0·1 *N* $CuSO_4$. Titrate with 0·01 *N* Na diethyldithiocarbamate till a stable yellowish-brown colour appears. Cations yielding insol. products with pyridine in the presence of EDTA can be masked with tartaric acid; Ag and excess of Bi interfere. The factor of the volumetric soln. must be checked frequently. J. ZÝKA

795. **Determination of substances in minute quantity. XIII. Quantitative determination of a small amount of mercury in the presence of a large amount of chloride ion and an equilibrium of "enolised" dithizone - mercury (II) compound.** T. Kato, S. Takei and A. Okagami. *Technol. Rep. Tôhoku Univ.*, 1957, **21** (2), 107–121.—Optimum conditions are established for the determination of small amounts of Hg in the electrolyte obtained in the commercial manufacture of soda. After addition of complexing agents to prevent interference from other metal ions and $NaHSO_3$ to destroy oxidising agents, the Hg - dithizone complex is extracted into CCl_4 and determined spectrophotometrically. The sample soln. containing 1 to 25 μg of Hg is brought to ≃ pH 5 with buffer soln. and a suitable amount of complexing agent is added. Zinc, Pb, Cd and up to 1 mg of Cu can be masked with EDTA; for quantities of Cu greater than 1 mg, addition of 1:2-diamino*cyclo*hexane-NNN′N′-tetra-acetic acid is recommended; $NaHSO_3$ is then added when oxidising agents are present. The soln. is shaken for 5 min. with 10 ml of 2 to 3×10^{-5} *M* dithizone. After the organic layer has been passed through filter-paper to remove emulsified water, its absorbance at 490 mμ is measured in 10-mm cells. A. JOBLING

796. **Spectrophotometric determination of boron with diaminochrysazin, diaminoanthrarufin and tribromoanthrarufin [4:5-diamino-1:8-, 4:8-diamino-1:5-, and 1:5:(?)-tribromo-4:8- dihydroxyanthraquinone].** E. C. Cogbill and J. H. Yoe (Pratt Trace Analysis Lab., Dept. of Chem., Univ. of Virginia, Charlottesville, U.S.A.). *Anal. Chem.*, 1957, **29** (9), 1251–1258.—Boron in the range 2 to 7 μg can be determined with a precision within 1% by the use of these reagents. All of them are used in H_2SO_4 and follow Beer's law up to 7 μg in 10 ml. The only common metal ion likely to interfere is Ti^{4+}; oxidising anions and F^- must be absent. In a procedure for determining boron in plant tissue, wet digestion is used and separation of boron by methyl borate distillation. If 4:5-diamino-1:8-dihydroxyanthraquinone is used, 6 to 12 μg of boron in a 0·1 to 0·3-g sample of plant tissue may be determined with a precision within 4%. K. A. PROCTOR

797. **Ultra-violet spectrophotometric determination of cerium(III).** H. L. Greenhaus, A. M. Feibush and L. Gordon (Dept. of Chem., Syracuse Univ., N.Y., U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1531–1534.—Tervalent Ce can be determined rapidly by measurement of its u.v. absorption at 253·6 mμ in *N* H_2SO_4. Beer's law is obeyed for 0 to 400 mg of Ce per litre. With 80 mg of Ce per litre, the method gave a 3% error with approx. 100 to 200 mg of Pr, Nd, Sm, Ga, Dy, Ho, Er, Yb or Y present. Europium and La interfere when in excess of 40 mg per litre; Hf, Zr, Th and Cu interfere more strongly; NO_3^- and UO_2^{2+} cannot be tolerated; Na, K and Cl^- do not interfere; Ce^{IV} interferes, but can easily be reduced to Ce^{III} with H_2O_2. The coeff. of variation for 10 determinations of a 0·1% soln. was $< \pm 1\%$. K. A. PROCTOR

798. **Chemical analysis on the microgram scale. V. Separation and identification of cerium, thorium, lead, calcium, strontium and barium.** J. K. Thompson and C. L. Wilson (Chem. Dept., Queen's Univ., Belfast). *Mikrochim. Acta*, 1957, (3–4), 334–340 (in English).—Modifications are made to the non-H_2S qual. analysis scheme (*Anal. Chim. Acta*, 1950, **4**, 440) devised for the milligram scale, so as to make it suitable for the microgram scale. J. H. WATON

799. **Analysis for industry [methods for determining carbon dioxide].** M. R. Earnshaw. *Ind. Chem. Mfr*, 1957, **33**, 253–255.—Gravimetric, titrimetric, gasometric and other methods for determining CO_2 are reviewed. (39 references.) S.C.I. ABSTR.

800. **The determination by radioactivation of trace quantities of arsenic, antimony and copper in pure silicon.** A. A. Smales, D. Mapper, A. J. Wood and L. Salmon (A.E.R.E., Harwell, England). A.E.R.E. Report C/R 2254, 1957, 26 pp.—The Si (300 to 400 mg) and standards are neutron-irradiated in a pile. After the surface has been cleaned with NaOH soln. and H_2O_2, the sample is fused with NaOH, KNO_3, As and Sb. The melt is dissolved in water, and $CuSO_4$ soln. and H_2O_2 are added. Copper and Sb are pptd. by adding $Na_2S_2O_4$ to the hot soln. Arsenic is recovered from the soln. by distillation with HBr, then pptd. with $NH_4H_2PO_2$ and counted. After purification, the Sb and Cu are pptd. as Sb_2S_3 and CuSCN, respectively, and counted. The contents of As, Sb, Cu, Ta and Zn of some samples were such that they could be determined directly by using a γ-spectrometer. As little as 0·001 p.p.m. of As, Sb and Cu could be measured. G. J. HUNTER

801. **Differential thermal analysis. New technique for testing silica - alumina catalysts.** R. L. Stone and H. F. Rase (Robert L. Stone Co., 2607 Hillview Rd., Austin, Tex., U.S.A.). *Anal. Chem.*, 1957, **29** (9), 1273–1277.—Samples of the catalyst and an inert material are exposed to an active gas, such as ammonia, under standard conditions. The difference in temp. rise between the two samples is measured by a differential thermal analyser, and is regarded as being proportional to the activity of the catalyst. G. S. ROBERTS

802. **Determination of traces of lead in igneous minerals.** A. D. Maynes and W. A. E. McBryde (Univ. of Toronto, Ontario, Canada). *Anal. Chem.*, 1957, **29** (9), 1259–1263.—A method is described for the determination of microgram quantities of Pb with dithizone. Interference from other metals, particularly Bi and Tl, is avoided by extraction of the Pb with a chloroform soln. of diethylammonium diethyldithiocarbamate. The method was used successfully with various minerals. A precision within 3% was attained for 17 to 2400 p.p.m. of Pb. G. S. ROBERTS

803. **Determination of lead dioxide in minium [red lead].** C. L. Laméris and D. W. Tempelman (Opleidingscentrum voor Tropische Landbouw,

Deventer, Holland). *Chem. Weekbl.*, 1957, **53** (35), 473.—Shake 1 g of dried red lead in a flask with ≃ 25 ml of HNO_3 (1:1, sp. gr. 1·2), if necessary with gentle heating, until the red colour has disappeared. Then add ≃50 ml of water and 20 ml of 0·2 *N* oxalic acid, again shaking until the brown ppt. of PbO_2 has dissolved; it may be necessary to warm the soln. and to crush any hard particles. Add ≃5 ml of 10% $Fe(NO_3)_3$ soln. and back-titrate with standard $KMnO_4$. A sample of red lead analysed by the iodimetric method gave a value of 30·2% of PbO_2 as compared with 30·1% by this oxidimetric procedure. P. HAAS

804. **Fluorimetric determination of tin with flavanol.** C. F. Coyle and C. E. White (Univ. of Maryland, College Park, U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1486–1488.—Variables in the determination are discussed. The complex, containing one atom of tin to one of flavanol, is prepared by treating the soln., containing 0·1 to 0·6 μmole of Sn^{IV}, with dil. H_2SO_4 and flavanol in the presence of dimethylformamide. The soln. is irradiated at 405 mμ and the fluorescence measured at 470 mμ. The concn. of Sn is derived from standards. Of several possible interferences that were investigated, only F^-, PO_4^{3-}, Zr^{4+}, Mo^{VI}, and high concn. of Al^{3+} or Th^{4+} cause errors. A sensitivity of 0·002 μmole per 10 ml of soln. is given. The reaction can be used qual. in the absence of dimethylformamide. D. A. PANTONY

805. **Determination of sulphur in titanium [and titanium alloys].** M. Codell, G. Norwitz and C. Clemency (Pitman-Dunn Lab., Frankford Arsenal, Philadelphia, Pa., U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1496–1499.—The sample containing up to 1% of S is dissolved in a mixture of HCl and HF contained in the distilling flask of a distillation absorption apparatus (described). The liberated H_2S is distilled in a stream of He and absorbed in ammoniacal $CdCl_2$. The soln. is then titrated with standard KIO_3 and the content of S calculated from a given equation. Precision and accuracy data are tabulated. If present in the metal, Se and As and possibly C would interfere. For very low concn. of S, absorption in aq. NH_3 and colorimetric measurement of the PbS after addition of Pb citrate is suggested. D. A. PANTONY

806. **Colour reaction between thorium and quercetin and separation scheme for interfering ions.** O. Menis, D. L. Manning and G. Goldstein (Analytical Chem. Div., Oak Ridge National Lab., Tenn., U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1426–1430.—The effect of variables, such as pH of the soln., interfering ions, and ratio of reagents, is investigated in the development of a spectrophotometric determination of Th^{4+} with quercetin. The recommended procedure is as follows. The soln., containing 10 to 150 μg of Th^{4+} and approx. 15 ml in vol., is adjusted to a pH of 2·8 to 3 with Na_2CO_3; the soln. is made up to standard vol. with ethanol, quercetin reagent and water. After 15 min. the absorption is measured at 422 mμ against a reagent blank. Interfering ions, especially PO_4^{3-}, are removed by a preliminary ion-exchange separation or by extraction of Th^{4+} with thenoyltrifluoroacetone at a pH of 2. Beer's law holds and the Th^{4+} concn. can be derived from standards or a calibration curve. The coloured complex is established as containing 2 mol. of quercetin to 1 mol. of Th, and has a dissociation constant of $1·2 \times 10^{-10}$. D. A. PANTONY

807. **Polarographic determination of sulphamic acid and nitrite.** J. Gottfried and J. V. A. Novák (Res. Inst. Org. Chem., Ústí n/Lab., Czechoslovakia). *Chem. Průmysl*, 1957, **7** (9), 476–478.—The polarographic titration of sulphamic acid with the use of a standard soln. of $NaNO_2$ in acid medium (0·1 *N* HCl) is described. The procedure is mainly suitable for the determination of small amounts of sulphamic acid in waste lyes which contain much H_2SO_4 and sulphates, and for the determination of nitrites in the presence of imidosulphonates; it has also been adapted for the determination of nitrite in the presence of nitrate. J. BÖSWART

808. **A selective test for the detection of orthophosphate.** J. W. Robinson and P. W. West (Esso Research Lab., Baton Rouge, La., U.S.A.). *Microchem. J.*, 1957, **1** (1), 93–99.—*o*-Dianisidine molybdate (**I**) is proposed as a new reagent for spot tests. *Reagent*—Dissolve $Na_2MoO_4.2H_2O$ (2·5 g) in 20 ml of 7·5% (v/v) H_2SO_4. Dissolve *o*-dianisidine (0·25 g) in 1 ml of conc. H_2SO_4 and add it slowly to the sodium molybdate soln., with stirring. Set the soln. aside overnight, filter, and discard the ppt. *Procedure*—On a white spot-plate place one drop of test soln., one drop of **I** and one or two drops of hydrazine hydrate. On the addition of **I**, a brown ppt. forms. On the addition of hydrazine hydrate the conversion of the brown ppt. to a molybdenum blue colour confirms the presence of PO_4^{3-}. The limit of detection is 0·05 μg of PO_4^{3-} or one drop of a soln. containing 1 μg per ml. The limiting dilution is 1 p.p.m. The only serious interference is sulphide ion, although some compounds mask the appearance of the ppt. formed at the end of the first step. D. F. PHILLIPS

809. **Rapid analytical methods for metals and minerals. III. Polarographic determination of vanadium in minerals.** Z. Šulcek (Central Geolog. Inst., Prague, Czechoslovakia). *Chem. Listy*, 1957, **51** (8), 1453–1456.—After fusion with Na_2CO_3, V can be separated from interfering elements and determined polarographically in 0·2 *M* KCN and 0·02 *M* EDTA. *Procedure*—Evaporate the sample (0·5 to 1 g) in a platinum crucible with a mixture of HF (40%) (5 to 10 ml) and H_2SO_4 (1:1) (0·5 ml) and fuse the residue with a mixture of Na_2CO_3 (6 g) and borax (0·5 to 1 g). Cool the melt and wash it with hot water (100 ml) into a beaker. Reduce the resulting manganate with Na_2O_3. Boil, filter and wash the residue on the filter with Na_2CO_3 soln. (0·5%). Add HCl to the filtrate, evaporate to 70 ml, and dilute to 100 ml with water. Measure 25 ml into each of two 50-ml flasks, add 0·2 *M* EDTA (disodium salt) (5 ml) and to one of the flasks 1 to 3 ml of 0·02 *M* NH_4VO_3. Neutralise the contents with NaOH (bromothymol blue indicator), add 2 *M* KCN (5 ml) and gelatin soln. (0·5%) (0·2 ml), adjust the vol. with water, remove oxygen and register the polarographic waves from − 0·8 V. The results were in good agreement with those of colorimetric determinations. J. ZÝKA

810. **Separation and determination of tantalum.** G. R. Waterbury and C. E. Bricker (Univ. of California, Los Alamos Sci. Lab., N.M., U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1474–1479.—Details are given of the dissolution of uranium alloys and plutonium alloys. An aliquot of the soln. containing 0·025 to 3 mg of Ta is evaporated to fumes of H_2SO_4. The residue is cooled, diluted and treated

with HF, and the TaF_7^{2-} are extracted with 4-methylpentan-2-one (hexone). The org. phase is evaporated in the presence of NaOH and, if Ti or Mo is present, the extraction and evaporation are repeated. The residue is dissolved in a little HF and the soln. evaporated to fumes with H_2SO_4. Org. material is decomposed with $S_2O_8^{2-}$ and the residue is transferred to a standard flask with conc. H_2SO_4. Quinol in H_2SO_4 is added and the soln. is made up to vol. Absorption is measured at 375 mμ against a reagent blank. The concn. of Ta is calculated from standards. Recovery is stated to be 100·2% with a coeff. of variation of ± 1·3% over the range 0·5 to 3·39 mg of Ta. Niobium is the only serious interference. D. A. PANTONY

811. **A field method for the rapid estimation of the oxides of niobium and tantalum in black-sand concentrates.** H. C. Curwen (Brit. Guiana Consolidated Goldfields, Ltd.). *Bull. Instn Min. Metall., Lond.*, 1956, (600), 39–41.—*Procedure*—Fuse the sample (0·2 to 0·3 g, 80 mesh) with $KHSO_4$ (3 to 5 g) in a silica crucible; when cool add 25% (w/v) tartaric acid (20 ml), warm to loosen the melt, and wash the contents of the crucible into a beaker with hot H_2O (40 ml). Add dil. H_2SO_4 (2 to 3 ml) and boil (if any blackish grains remain Sn may be present); collect and wash the residue, and to the filtrate add dil. HCl (50 ml) and then boil the soln. for ≃ 5 min. (hot-plate) to precipitate the bulk of the Nb and Ta. Collect and wash the ppt. thoroughly with hot dil. HCl, and finally ignite it strongly and weigh as $(Nb,Ta)_2O_5$. The error is ≃ − 5%; ≃ 16 samples can be analysed per day by one analyst and assistant. Tungsten should be removed by preliminary washing of the final ppt. with warm dil. aq. NH_3. Other interfering metals can be removed by an initial treatment of the sample with acid (boil, filter, wash) before the fusion. W. J. BAKER

812. **Reaction of oxygen in a mass spectrometer to form carbon monoxide.** G. F. Crable and N. F. Kerr (Gulf Res. & Development Co., Pittsburgh, Pa., U.S.A.). *Anal. Chem.*, 1957, **29** (9), 1281–1282.—The reaction of oxygen with carbonaceous materials has been studied in the ion chamber of a mass spectrometer. The production of CO is directly proportional to the oxygen partial pressure, and is believed to be formed by oxygen decarbiding the tungsten filament. K. A. PROCTOR

813. **Spectrographic determination of oxygen in metals.** R. W. Tabeling (Iowa State Coll., Ames, U.S.A.). *Iowa St. Coll. J. Sci.*, 1957, **31** (3), 528–529.—A steel sample (1 g) was fused in a carbon electrode at 20 amp. Any oxides present were reduced to give CO which was liberated into the argon atmosphere of the chamber. The intensity ratio of O_{7771} to A_{7891} was measured to determine the amount of oxygen in the original sample. The sensitivity was approx. 0·0005%, and the average precision 10% over the concn. range of 0·002% to 0·06% of oxygen in steel. The analysis took about 15 min. Oxygen was successfully determined in nickel by a similar spectrographic method. The oxygen blank arising from absorbed gases in the carbon electrodes was eliminated by a preliminary outgassing at 1000° in high vacuum. In the determination of oxygen in lanthanum, a nickel bath was used in reduction of the oxides. The whole assembly was placed in a carbon electrode and arced at 30 amp. O. M. WHITTON

814. **Analysis of elementary sulphur in acetone solution.** Wasaburo Jono, Teizo Watanabe, Tokio Rokushima and Hajime Maruo (Fac. of Engng, Kobe Univ., Nagata-ku, Kobe). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1957, **60** (4), 502–503.—The $E_{\frac{1}{2}}$ value of S in acetone (≃ 0·01 *M*) remains const. on varying the amount of methanol in the basal soln. containing acetate buffer of pH 5 to 6·5, but the wave height increases with increase in methanol content. Addition of gelatin has little effect on the wave height, but shifts the $E_{\frac{1}{2}}$ value in the positive direction by 0·015 to 0·025 V. The wave height is proportional to the concn. of S for < 0·01 *M* S in acetone at < 30°, in a soln. containing 3 vol. of methanol, 1 vol. of the acetone soln. of S, 1 vol. of the acetate buffer and 0·1 vol. of 0·5% gelatin. Oxygen must be completely removed. K. SAITO

815. **Rapid and accurate determination of sulphur in pyrites by the combustion method.** A. Mendelowitz (Res. Dept., African Explosives and Chem. Ind., Ltd., P.O. Northrand, Transvaal, S. Africa). *J. S. Afr. Chem. Inst.*, 1957, **10** (1), 36–39.—The sample is mixed with kieselguhr (to prevent sintering) and burnt in a stream of O at ≃ 950°. The resulting gases are passed through (*i*) neutral H_2O_2 and (*ii*) a dry asbestos filter (to retain acid "mist"). The contents of both (*i*) and (*ii*) and all connections are washed into a beaker and titrated with 0·1 *N* $Na_2B_4O_7$. For a content of S of ≃ 40%, the standard deviation is < 0·10. S.C.I. ABSTR.

816. **Improvements in or relating to the detection and estimation of sulphur dioxide in a gaseous mixture.** S. A. des Manufactures des Glaces et Produits Chimiques de Saint-Gobain, Chauny et Cirey. [Inventor: C.-M. Cherrier]. Brit. Pat. 771,531; date appl. 20.3.52.—The invention relates to a method of measuring the u.v. absorption, at 305 to 320 mμ or 265 to 285 mμ, of a current of gas flowing through a conduit. N. E.

817. **Spectro - visual method for determining end-points. Application to titration of soluble sulphate.** H. H. Bovee and R. J. Robinson (Univ. of Washington, Seattle, U.S.A.). *Anal. Chem.*, 1957, **29** (9), 1353–1355.—Improved detection of the end-point of the titration of SO_4^{2-} by $BaCl_2$ soln. is claimed. Tetrahydroxybenzoquinone is used as indicator and the titration soln. is illuminated by the light from 15-W fluorescent lamps after passage through a Wratten filter No. 45. The end-point is taken at the colour change of green to blue. An accuracy of the order of ± 0·4% can be expected. The presence of PO_4^{3-}, F^- or NO_3^- causes appreciable interference, but these anions may be removed by an ion-exchange technique (Fritz and Yamamura, *Ibid.*, 1955, **27**, 1461, and Fritz *et al.*, *Ibid.*, 1957, **29**, 158). G. S. ROBERTS

818. **Infra-red spectrophotometry of sulphate ion. Combining freeze-drying with potassium bromide disc technique.** Han Tai and A. L. Underwood (Dept. of Chem., Emory Univ., Ga., U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1430–1433.—The application of i.r. analysis to aq. soln. of inorganic samples is described. In determining the SO_4^{2-}, the optimum analytical range is 30 to 80 μg with coeff. of variation of ± 2·3%. K. A. PROCTOR

819. **Semi-quantitative estimation of dithionite.** T. P. Whaley and J. A. Gyan (Res. Lab., Ethyl Corp., Detroit, Mich., U.S.A.). *Anal. Chem.*, 1957,

29 (10), 1499.—A procedure is described in which the sample soln. is treated with naphthol yellow S in dil. aq. NH_3: on mixing, the colour changes immediately from yellow to red, and the time taken for the yellow colour to reappear is a semi-quantitative measure of the $S_2O_4^{2-}$ concn. Sulphite, alkoxide, Cl^-, SO_4^{2-}, OH^-, S^{2-} and SO_2 do not interfere. D. A. PANTONY

820. **New colour test for selenium.** E. Sawicki (Taft San. Engng Center, Cincinnati, Ohio, U.S.A.). *Anal. Chem.*, 1957, **29** (9), 1376–1377.—An aq. soln. of 4-methylthio-1:2-phenylenediamine hydrochloride is mixed with the test soln. containing Se in the form of selenous acid. A drop of the mixture is evaporated to dryness and redissolved in conc. H_2SO_4, when a blue - purple colour is formed. The limit of identification is 0·05 μg of Se, and the limiting concn. is 1 in 4×10^6. The selectivity of the reaction was not investigated.

G. S. ROBERTS

821. **Separation of tellurium, ruthenium, caesium and rare earths from one another, using anion-exchange resin Dowex-1.** M. V. Šušić (Inst. Nucl. Sci., Belgrade). *Bull. Inst. Nucl. Sci., Belgrade*, 1957, **7**, 39–41 (in English).—Rare-earth elements can be separated from Te, Ru and Cs by making the soln. alkaline (KOH) and then heating to precipitate the bulk of the rare earths as hydroxides. The cool filtrate (pH > 7) is passed through a column (10 cm $\times$ 0·8 cm) of Dowex-1 resin, whereby Te and Ru are quant. adsorbed while Cs, K and trace amounts of rare-earth elements pass through completely. Tellurium and part of the Ru can be eluted from the column with HCl (1 + 10), whilst the remainder of the Ru can then be eluted slowly with conc. HCl. The concn. of each element should be $\simeq 10^{-2}$ to 10^{-5} g per 10 to 25 ml.

W. J. BAKER

822. **Extraction and flame-spectrophotometric determination of chromium.** H. A. Bryan and J. A. Dean (Univ. of Tennessee, Knoxville, U.S.A.). *Anal. Chem.*, 1957, **29** (9), 1289–1292.—The Cr in samples of alloys or minerals is dissolved by acid digestion, and converted into $Cr_2O_7^{2-}$ by oxidation with potassium peroxydisulphate in the presence of Ag^+. The $K_2Cr_2O_7$ is then extracted with 4-methylpentan-2-one, the organic phase is aspirated into an oxy-acetylene flame, and the characteristic line emissions of Cr are measured. The method can be used in the presence of large amounts of Fe^{III}, which is the only element that may be extracted with Cr. Concn. of Cr as low as a few tenths of a microgram per ml can be determined by this method.

G. S. ROBERTS

823. **Radiometric titration with silver and cobalt tungstates.** D. H. Bradhurst, B. A. W. Coller and J. F. Duncan (Chem. Dept., Univ., Melbourne, Australia). *J. Inorg. Nuclear Chem.*, 1957, **4** (5–6), 379–381.—Soln. of tungstate have been radiometrically titrated with Co and Ag labelled with ^{60}Co and ^{110}Ag, respectively. Cobalt tungstate pptd. rapidly from the boiling soln. at a pH $> 6·5$ and contained 23·5 $\pm$ 1·5% of Co. The composition of the silver tungstate varied with pH over the range 3·3 to 7·2. G. J. HUNTER

824. **Polarographic behaviour of the uranyl - cupferron system.** P. J. Elving and A. F. Krivis (Univ. of Michigan, Ann Arbor, U.S.A.). *Anal. Chem.*, 1957, **29** (9), 1292–1297.—Unusual polarographic phenomena found in soln. containing both U^V and cupferron have been studied in detail. At low concn. of U and cupferron, two waves appear, corresponding to U^{VI} - U^V and cupferron - phenylhydrazine reductions. The variations in height and $E_{\frac{1}{4}}$ of these waves are related to the formation, at the mercury surface, of an insol. film of U^{VI} cupferrate, which hinders the approach of uranyl ions to the electrode. K. A. PROCTOR

825. **Estimation of uranium content in rocks.** V. Cirilli (Ist. Chim. Generale ed Appl. e Metall., Politec. Torino). *Ric. Sci.*, 1957, **27** (3), 674–683.—A brief description is given of some polarographic, spectrophotometric and fluorimetric methods for determining U. If the polarography is carried out in the presence of citric acid, 0·1 to 0·2% of U can be determined in a mineral containing 10% of Fe. Phosphates interfere. In the spectrophotometric method, the pH of the soln. should be above 11; V and Fe interfere. The use of the fluorimetric method enables amounts of U of the order of 10^{-10} g to be detected. The sample soln. is freed from interfering elements by paper chromatography, use of organic selective solvents, or ion-exchange resins.

L. KRUSZYNSKA

826. **Identification of fission products of uranium by means of paper chromatography.** H. Götte and D. Pätze (Max Planck Inst. für Chem., Mainz, Germany). *Angew. Chem.*, 1957, **69** (18–19), 608–614.—A simple analytical scheme is given for the qual. paper chromatography of radioactive isotopes having half-lives of > 5 hr. The most important fission products of uranium irradiated for periods from 6 days to 3 months can be separated and identified by three chromatograms with such solvents as methanol, ethanol, butanol and HCl. The spots are revealed chemically and by autoradiography. The procedure is similar to that described previously (*cf. Z. Elektrochem.*, 1954, **58**, 636), except that provision is made for the separation and identification of those elements that can be pptd. with H_2S. W. J. BAKER

827. **Polarographic - amperometric determination of mercury, silver, gold, iron and vanadium. Determination of vanadium in uranium.** M. V. Šušić (Inst. Nucl. Sci., Belgrade). *Bull. Inst. Nucl. Sci., Belgrade*, 1957, **7**, 25–28 (in English).—Silver, Hg^{2+}, Au^{3+}, Fe^{3+} and V^{5+}, in concn. (C) of $\simeq 10^{-3}$ to 10^{-6} g per ml, can be determined polarographically in the presence of a non-complexing electrolyte, *e.g.*, HCl, H_2SO_4, $(NH_4)_2SO_4$ or K_2SO_4. The diffusion current (I_d) of the sample soln. alone is measured first, a known vol. of standard soln. of the ion being determined is added and I_d is again measured. Zero diffusion current is obtained in each instance by adding an excess of ascorbic acid as reductant. The plot of I_d *vs.* C is linear. This procedure can be used to determine V^{5+} in soln. containing up to a 200-fold excess of U; the supporting electrolyte is $(NH_4)_2SO_4$ slightly acidified with H_2SO_4 and I_d is measured from + 0·1 to 0·0 V *vs.* the S.C.E.

W. J. BAKER

828. **Separation of uranium from rare earths and other elements by solvent extraction with tributyl phosphate.** M. V. Šušić and N. Jelić (Inst. Nucl. Sci., Belgrade). *Bull. Inst. Nucl. Sci., Belgrade*, 1957, **7**, 29–33 (in English).—A separation of U from rare-earth elements (*e.g.*, Ce and Eu), or from Zn, Cd, Fe, Bi, Pb, Cu and Co, in 2 N HNO_3 soln. can be made by extraction of U into a 20% (v/v) soln.

of tributyl phosphate (**I**) in kerosene, followed by its re-extraction into 2 *N* H_2SO_4. The influence of the distribution coeff. of U, rare-earth and other elements on the efficiency of extraction into **I** is discussed. Max. extraction of U is attained by keeping the concn. of U as high as possible, *e.g.*, 10^{-3} to 10^{-4} g per ml; the vol. of the aq. and organic phases should be approx. equal.
W. J. BAKER

829. **Separation of uranium from rare earths and other elements in sulphuric acid medium, using anion-exchange resin.** M. V. Šušić (Inst. Nucl. Sci., Belgrade). *Bull. Inst. Nucl. Sci., Belgrade*, 1957, **7**, 35–38 (in English).—Uranium can be separated from rare earths (*e.g.*, ^{144}Ce), alkali metals (*e.g.*, ^{137}Cs) and other elements (Cu, Fe, Zn, Cd, Mn, Co, Ni) in 0·01 *N* H_2SO_4 soln. by quant. adsorption on an anion-exchange column (Dowex-1, 10 cm × 0·8 cm), followed by elution with *N* HNO_3. All the other elements pass through the column unadsorbed. The concn. of each element should be $\simeq 10^{-2}$ to 10^{-4} g per 20 ml, and the rate of flow $\simeq$ 1 ml per min. per sq. cm. W. J. BAKER

830. **Influence of temperature on the extraction of uranyl nitrate with tributyl phosphate at varying concentrations of nitric acid.** Z. I. Dizdar, O. S. Gal and J. K. Rajnvajn (Inst. Nucl. Sci., Belgrade). *Bull. Inst. Nucl. Sci., Belgrade*, 1957, **7**, 43–48 (in English).—In the system aq. HNO_3 - 30% tributyl phosphate (diluted with dibutyl ether), the mass partition coeff. (f) of $UO_2(NO_3)_2$ decreases with increasing temp. (T = 1° to 80°) at all concn. of HNO_3 up to 10 *M*, but for a specific concn. of HNO_3 f is proportional to log T. As indicated by the changes in vol. and optical transmission, and by the decrease in acidity of the aq. phase, the degree of chemical reaction between the two phases increases with T and acid concn. The limit of stability is 6 *M* HNO_3 at 20° and 4 *M* HNO_3 at 80°. At 20° the optimum acid concn. for the formation of the uranyl dinitrato complex is 5 *M*. W. J. BAKER

831. **Separation of cerium and europium from uranium by organic co-precipitants.** Z. B. Maksimović (Inst. Nucl. Sci., Belgrade). *Bull. Inst. Nucl. Sci., Belgrade*, 1957, **7**, 49–52 (in English).—The separation is based on the quant. co-pptn., at pH 8 to 9, of Ce and Eu with a 2% aq. soln. of 4:4′-di-(4-hydroxy-3-sulphophenylazo)diphenyl (**I**) and a 1% aq. soln. of methyl violet (**II**) in the presence of ammonium carbonate to complex the U. The addition of **I** is essential for the formation of silky crystals without which the quant. co-pptn. (on addition of **II**) cannot take place. Thus, in the concentration of nuclear impurities for spectrography, $\simeq 10^{-9}$ g of ^{144}Ce and ^{152}Eu per ml can be separated from 1 g of U, only 1 to 2 mg of U being co-pptd. W. J. BAKER

832. **Coulometric determination of plutonium.** W. N. Carson, jun., J. W. Vanderwater and H. S. Gile (Hanford Atomic Products Operation, Gen. Elect. Co., Richland, Wash., U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1417–1422.—Plutonium samples, containing 0·003 to 10 mg of the element, are treated with dil. $KMnO_4$, HNO_3 and $HClO_4$ and the soln. is stirred with an inert gas stream at 75°. When oxidation to Pu^{VI} is complete, dil. formaldehyde soln. is added to remove all higher valencies of Mn. The soln. is then titrated in a coulometric titration apparatus (described) with electrolytically generated Fe^{2+} by which the Pu^{VI} is quant. reduced to Pu^{IV}, although response is slow near the end-point. The concn. of Pu is calculated from a given equation. Details are given for pretreatment of samples containing interfering elements, and of these only the interference due to Ce^{IV} is not overcome. Precision is given as 5% at the 3-μg level, and 1% at the 10-mg concentration.
D. A. PANTONY

833. **The effect of oxygen isotopes in the analysis of plutonium isotopes using plutonium oxide ion beams in the solid source mass spectrometer.** G. R. Hall and G. H. Palmer (A.E.R.E., Harwell, England). A.E.R.E. Report C/R 1150, 1957, 5 pp.—Equations are developed that enable the recorded intensities of the PuO^+ and PuO_2^+ beams to be corrected for the presence of ions containing ^{17}O and ^{18}O. G. J. HUNTER

834. **Spectrophotometric studies of plutonium in nitric acid solution. I.** G. R. Hall, P. D. Herniman and A. J. Walter (A.E.R.E., Harwell, England). A.E.R.E. Report C/R 712, 1957, 22 pp.—The preparation of solutions of Pu^{3+}, Pu^{4+} and Pu^{6+} in HNO_3 is reported and the available methods for the preparation of these solutions and that of Pu^{5+} are reviewed. The absorption spectra peaks for Pu^{3+} in 0·6 *M* HNO_3 at 564 and 602 mμ, Pu^{4+} in 0·6 *M* and 5 *M* HNO_3 at 476 and 600 mμ, Pu^{5+} at 569 and 775 mμ, and Pu^{6+} in 0·25 *M* HNO_3 at 832, 952 and 982 mμ are discussed. Investigation of the sharpness of the 832-mμ Pu^{6+} band leads to the conclusion that calibration of the absorption assay method under the exact experimental conditions of the unknown determination is essential if large errors are to be avoided. E. G. CUMMINS

835. **Analysis of refractory oxides for halogens.** M. P. Chaika. *Optika i Spektrosk.*, 1957, **2** (4), 421–425.—Details are given of the spectroscopic analysis of F and Cl in U_3O_8 and ThO_2 in which the spectra are excited in a hollow iron cathode with helium as the carrier gas. The influence of the cathode dimensions on halide evolution is given in tables. It is claimed that a concn. of 10^{-4} % of F in U_3O_8 may be estimated with 10% error on a minimum 10-mg sample. PHYS. ABSTR.

836. **Comparison of spectrophotometric and visual titration for the determination of fluorine.** J. A. Dean, M. H. Buehler and L. J. Hardin (Univ. Tennessee Agric. Exp. Sta., Knoxville, U.S.A.). *J. Ass. Off. Agric. Chem.*, 1957, **40** (3), 949–958.—Results obtained for the determination of F in samples of urine, grain, bone and lucerne, by the use of the visual titration of F^- with thorium nitrate, are in general agreement with those afforded by spectrophotometric titration at 540 mμ. Visual titration by an experienced operator is much the more rapid procedure. A. A. ELDRIDGE

837. **Acidimetric determination of fluorine after ion exchange. Application to aluminium fluoride, cryolite and fluorspar.** H. Shehyn (Aluminium Lab., Ltd., Arvida, Quebec, Canada). *Anal. Chem.*, 1957, **29** (10), 1466–1470.—The fluorine in the minerals is converted into alkali fluorosilicate by means of fusion with Na_2CO_3 - K_2CO_3 mixture in the presence of SiO_2. After extraction with water and filtration, the soln. is treated with a cation-exchange medium (Amberlite IR-120). The eluate is boiled to remove CO_2 and is then treated with excess of $CaCl_2$; the liberated HCl is titrated while

hot with NaOH, with bromocresol purple as indicator. Fluorine contents are calculated from given formulae. Accuracy and precision data are presented and compared with those from other procedures. D. A. PANTONY

838. **Test for fluoride in calcium phosphate.** G. J. Mulder and E. M. Aarts (Farm. Lab. Rijksuniv., Utrecht, Holland). *Pharm. Weekbl.*, 1957, **92** (19), 661–666.—Vogelenzang's limit-test has been checked for the 6th edition of the Pharmacopoeia of the Netherlands. The distillation yield of the fluoride has been improved (90%) by using a 100-ml distillation flask, doubling the amount of water and sulphuric acid and distilling off 20 ml rather than 10 ml. An automatic shaker is recommended. The interference of $\simeq 4{\cdot}2$ mg of SO_4^{2-} and < 5 μg of PO_4^{3-} is negligible. P. RENTENAAR

839. **Determination of total oxidising power of buffered chlorous acid solutions.** Yoshio Tomimatsu and H. F. Launer (Western Utilization Res. Branch, Agric. Res. Service, U.S. Dept. Agric., Albany 10, Calif., U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1500–1501.—Chlorate alone may be determined by treating the soln. with standard Fe^{2+} in the presence of a NaH_2PO_4 - H_3PO_4 buffer and O is expelled by means of CO_2 derived from added $NaHCO_3$. After addition of H_2SO_4, the mixture is heated, cooled and then back-titrated with standard $KMnO_4$ soln. Correction is made for a blank. When ClO_2^- and ClO_2 are also present a similar procedure may be adopted except that great care is taken to avoid loss of ClO_2. Alternatively, Fe^{2+} may be replaced by As_2O_3 and $KMnO_4$ by $Ce(SO_4)_2$. Precision data for "total oxidising power" are presented: a coeff. of variation of $\pm$ 0·13% at 0·22 oxidising milli-equiv. is quoted. Reference is made to the determination of individual oxychlorine compounds. D. A. PANTONY

840. **Studies on the metal complexes of hydroxamic acids. I. Spectrophotometric determination of manganese with nicotino- and *iso*nicotino-hydroxamic acids.** R. L. Dutta (Indian Ass. for the Cultivation of Sci., Calcutta). *J. Indian Chem. Soc.*, 1957, **34** (4), 311–316.—Use is made of the red - violet colour given by Mn with nicotino- and *iso*nicotino-hydroxamic acids. The colour intensity is read at 480 to 490 mμ and 470 to 480 mμ, respectively. Beer's law is obeyed. Interference is caused by Cu, Ni, Co, Ag and Fe, but that of the first four metals can be suppressed with cyanide. C. A. SLATER

841. **Voltammetry and amperometric titration at the rotated platinum electrode of manganese(II), (III) and (VII) in pyrophosphate medium.** I. M. Kolthoff and E. Jacobsen (Univ. of Minnesota, Minneapolis, U.S.A.). *Microchem. J.*, 1957, **1** (1), 3–16.—The results of an extensive study of current - voltage curves of $KMnO_4$ and Mn^{III} in pyrophosphate medium at various pH values, with a platinum-wire electrode (sensitivity, 140 μA per millimole of Ag per litre) rotated at 900 r.p.m., are described. At a pH < 3, $KMnO_4$ in $Na_4P_2O_7$ buffer gave two waves, the first corresponding to a four-electron reduction to Mn^{III} and the second to a one-electron reduction. Between pH 4 and 8 only the four-electron reduction wave was observed. With Mn^{III} buffered at pH 3 or < 3 there was a well defined one-electron reduction wave, but no reduction was observed at a pH of 4 or > 4. Bivalent Mn can be determined by oxidation with lead dioxide in a pyrophosphate buffer followed by amperometric titration of Mn^{III} in the filtrate with ferrous iron at pH 0·5 at a potential of $-$ 0·2 V (*vs.* the S.C.E.) in the absence of air when the diffusion current of the Mn^{III} complex is measured. Alternatively, the titration may be carried out at $+$1·10 V when the anodic diffusion current of excess of ferrous iron is observed. The direct amperometric determination of Mn^{II} with permanganate is more convenient in a pyrophosphate buffer at pH 4 to 8 in the presence of air, the indicator electrode being short-circuited with the S.C.E. As little as 0·01 mg of Mn in a volume of 50 ml was titrated with an accuracy of $\pm$ 0·5%. Tervalent Ce and V^{IV} can be titrated in the same way and are separated from Mn by the pyridine procedure. No interferences from quite large amounts of vanadate, Ce^{IV} or chromate were observed. D. F. PHILLIPS

842. **New method of separating and determining rhenium.** V. Patrovský (Cent. Geolog. Inst., Prague, Czechoslovakia). *Chem. Listy*, 1957, **51** (7), 1295–1299.—A method for the separation of Re from various elements (mainly Mo) and optimal conditions for the photometric determination of Re with SCN^- in aq. or ether medium (without previous reduction) are described. *Procedure for molybdenite*—Fuse the sample (1 to 2 g) in an iron crucible with NaOH (5 to 8 g) and then with Na_2O_2 (5 g). Dissolve the melt in H_2O, filter, and wash the residue with H_2O. To the filtrate add HCl and make slightly alkaline with aq. NH_3. Add $ZnCl_2$ soln. (5%) (1 ml) and pass H_2S. Filter off the ppt., wash it with $(NH_4)_2S$ soln. (1%) then dissolve it in HCl (2 to 5 ml) with the addition of a few drops of H_2O_2, the excess being removed by boiling. Add $FeCl_3$ soln. (5%) (1 ml), conc. HCl (5 ml) and a soln. of SCN^- (20%) (5 ml). Dilute to between 60 and 80 ml and extract with ether (2 $\times$ 20 ml) (the aq. layer remains coloured). Add $SnCl_2$ soln. (50%) till the aq. layer becomes colourless, then add a few drops of $SnCl_2$ soln. in excess. Add conc. HCl (1 to 3 ml) and the same amount of SCN^-; extract with ether (2 $\times$ 10 ml), combine the extracts in a 25-ml flask, make up to vol. with ether, mix, and measure the extinction at 430 mμ. As little as 10^{-4} % of Re can be determined in a 1 to 2-g sample. When determining Re in manganese dioxide, silicates or iron ores, the sample must be dissolved in conc. HCl and H_2O_2 (or HNO_3) and the undissolved residue filtered off. An excess of Cu or Sb can be separated by reduction with metallic iron. J. ZÝKA

843. **Stabilisation of ferric thiocyanate colour in aqueous solution. Spectrophotometric method using ethyl methyl ketone.** P. Baily (Transvaal and Orange Free State Chamber of Mines, Richmond, Johannesburg, S. Africa). *Anal. Chem.*, 1957, **29** (10), 1534–1536.—The soln. of Fe^{3+} (10 to 200 p.p.m.) is made up to standard vol. with, successively, water, dil. HCl to adjust the pH to $\simeq 1{\cdot}5$, acetone, ethyl methyl ketone, aq. KSCN and finally water. The absorption is measured at 490 mμ. The colour is stable to light for at least 60 min. and the intensification of the colour by this method renders it more sensitive. Variables in the expt. are discussed. Of the ions tested, UO_2^{2+} and $P_2O_7^{4-}$ interfere. D. A. PANTONY

844. **Improved titrimetric determination of boron in ferrous metals.** R. Brühlmann and L. Piatti (Geb. Sulzer, A.-G., Winterthur, Switzerland).

Chimia, 1957, **11** (7), 203–205.—About 10 g of the sample (more or less according to the content of B) is dissolved in 60 ml of aq. HCl (2:1) under reflux. Hydrogen peroxide (15%) (30 ml) is added while cooling and the soln. is again boiled under reflux. After adding zinc chloride (10 g), water is distilled off until the temp. reaches 120°, the distillate being collected in 10 ml of H_2O containing 2 g of NaOH and cooled in ice. The residue is cooled to 70° and the usual determination of B by distillation with methanol is carried out; the titration of the methyl borate in the presence of mannitol has been improved by using a pH meter for the indication of the end-point. The alkali borate soln. remaining after the removal of methanol is neutralised with dil. HCl to a pH of 7·50. Acid-free mannitol is then added and the soln. is titrated with 0·02 *N* NaOH until the pH is again 7·50. By means of this improvement, both the accuracy and the range of determinable B are increased. L. S. ADLER

845. **Use of anion exchangers in analytical chemistry. II. Separation of small amounts of cobalt from nickel.** K. Liška and L. Klír (Res. Inst. Metals, Panenské Břežany, Czechoslovakia). *Chem. Listy*, 1957, **51** (8), 1547–1548.—With the use of the strongly basic anion exchanger OAL the separation of Co from Ni can be carried out. The separated Co can be determined colorimetrically by the use of furil α-monoxime or 1-nitroso-2 naphthol. The method is not convenient for complicated mixtures since Fe, Cu, Pb, Zn, Cd, Sn and other elements interfere. J. ZÝKA

846. **Spectrophotometric determination of ruthenium with 1:10-phenanthroline.** C. V. Banks and J. W. O'Laughlin (Inst. for Atomic Res., Iowa State Coll., Ames, U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1412–1416.—The sample soln., containing 10 to 150 μg of Ru and no strong oxidising agents, is evaporated to fumes with H_2SO_4. The residue is distilled in the presence of $HClO_4$ in a stream of air, the distillate, containing RuO_4, being collected under an aq. soln. of HCl and hydroxylamine hydrochloride. The receiving soln. is then evaporated and treated with dil. 1:10-phenanthroline reagent with further additions of hydroxylamine hydrochloride and adjustment of the pH to 6·0 with HCl or NaOH. After dilution to standard vol. and a development time of 2 hr., the absorption is measured at 448 mμ against a reagent blank. The effects of variables on the determination are discussed and displayed graphically. The sensitivity is given as 0·02 p.p.m. and the mean percentage deviation is $\simeq$ 1% in concn. of 0·1 to 1 μg per ml. D. A. PANTONY

847. **Polarographic studies of some precious metals. VIII. Oscillographic behaviour of some platinum-group metals and of gold.** P. Beran and J. Doležal (Inst. Anal Chem., Charles' Univ., Prague, Czechoslovakia). *Chem. Listy*, 1957, **51** (7), 1289–1294.—The oscillopolarographic behaviour of Pd, Ir, Rh, Pt, Au and Ag in various supporting electrolytes was studied. The depolarisation potentials of these metals were measured and the sensitivity of their oscillographic detection is given. In a medium containing HCl, even traces of Rh can be detected. J. ZÝKA

848. **Electro-chromatography in the separation of inorganic ions. Separation of precious metals.** A. K. Majumdar and M. M. Chakrabartty (Jadavpur Univ., Calcutta, India). *Naturwissenschaften*, 1957, **44** (1), 9.—Distinct separation of four ions in a mixture of the metals Pd^{II}, Pt^{IV}, Ir^{V}, Rh^{III}, Ru^{III}, Os^{IV} and Au^{III} is obtained with 0·1 *N* soln. of electrolyte with a constant voltage of 150 V for 5 hr. A list of suitable electrolyte soln. is presented. E. KAWERAU

849. **Spectrographic evaluation of separation of platinum from palladium, rhodium and iridium.** G. H. Ayres and H. J. Belknap (Dept. of Chem., Univ. of Texas, Austin, U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1536–1539.—The separation of Pt from Pd, Rh and Ir by double pptn. of the unwanted elements as their hydrous oxides has been shown, spectrographically, to be 99·94% complete, except in samples in which $<$ 20 mg of the elements was present. In the spectrographic determination, a d.c. arc was used with Co^{II} added as the chloride, as an internal standard. K. A. PROCTOR

See also Abstracts 767, 885, 1054, 1067, 1068.

3.—ORGANIC ANALYSIS

850. **Quantitative organic analysis. XII. Statistical evaluation of some methods of organic elementary analysis.** M. Večeřa and D. Šnobl (Res. Inst. Org. Synth., Pardubice-Rybitví, Czechoslovakia). *Chem. Listy*, 1957, **51** (8), 1482–1492.—The commonly used methods for the microdetermination of C, H, N, S and Cl have been evaluated statistically. J. ZÝKA

851. **Quantitative organic analysis. XI. The use of cobalto-cobaltic oxide as a combustion catalyst in elementary analysis. Preliminary communication.** L. Synek and M. Večeřa (Res. Inst. Org. Synth., Pardubice-Rybitví, Czechoslovakia). *Chem. Listy*, 1957, **51** (8), 1551–1552.—It has been found that cobalto-cobaltic oxide can be used as an effective combustion catalyst, even for difficultly combustible compounds. The activity of the catalyst does not change when used for the combustion of compounds containing sulphur or halides. *Preparation of the catalyst*—To asbestos (15 g) and $Co(NO_3)_2.6H_2O$ (10 g) add water (50 ml) and aq. NH_3 (5 drops), evaporate to dryness and ignite at 500°. J. ZÝKA

852. **Direct determination of oxygen in organic compounds.** I. Sheft and J. J. Katz (Argonne Nat. Lab., Lemont, Ill., U.S.A.). *Anal. Chem.*, 1957, **29** (9), 1322–1325.—Oxygen is quant. obtained from organic compounds by heating them at 500° with BrF_2SbF_6; vigorous shaking of the reaction vessel is necessary to complete the reaction. The liberated O is measured tensimetrically. The method is applicable to alcohols, aliphatic and aromatic acids, ethers, ketones, phenols, phosphate esters and sulphones, and to nitrogen-containing organic compounds in which the O is not bound to the N. The accuracy is within $\pm$ 1% and a precision within $\pm$ 0·2% was obtained from five replicate analyses. G. P. COOK

853. **A rapid micro Dumas method for nitrogen determination.** G. M. Gustin (Celanese Corp. of America, Summit, N.J., U.S.A.). *Microchem. J.*, 1957, **1** (1), 75–87.—The techniques and apparatus described are designed to remove all causes of a gas blank and hence the gas blanks themselves,

other than a constant adsorbed air blank from copper oxide. Shelburg's two-unit combustion train (*Anal. Chem.*, 1951, **23**, 1492) has been modified so that the reduced copper and hopcalite may be maintained at constant temp. in the second unit. This second unit can be isolated from the first unit after the completion of the combustion and sweep, permitting the next sample to be immediately set up. Zimmerman's gas reservoir (*Mikrochemie*, 1943, **31**, 42) was modified and placed next to the nitrometer to receive the gases swept from the combustion tube, as well as to buffer the uncontrolled evolution of gases during combustion. The mercury nitrometer, which was designed to permit immediate and easy measurements of the vol. of N, has a capacity of 1·3 ml. With organic samples of 1 to 10 mg a complete analysis takes 12 min. or less. The complete pyrolysis of all types of material is ensured by operating the furnace at 850°. The coeff. of variation based on 20 results was 0·2%. D. F. PHILLIPS

854. **Quantitative organic analysis. X. Micro-determination of chlorine and bromine in organic compounds.** M. Večeřa and J. Bulušek (Res. Inst. Org. Synth., Pardubice-Rybitví, Czechoslovakia). *Chem. Listy*, 1957, **51** (8), 1475–1481.—A new rapid micro-method is described. The sample is burnt in a stream of O and the resulting chloride is determined by an indirect neutralisation titration and the bromide argentimetrically with Brilliant yellow as indicator. J. ZÝKA

855. **Mineralisation of organic compounds with magnesium. V. Colorimetric micro-determination of phosphorus in organic compounds.** M. Jureček and J. Jeník (Inst. Anal. Chem., Pardubice, Czechoslovakia). *Chem. Listy*, 1957, **51** (7), 1312–1315.—When organic compounds containing P are burned with metallic magnesium, the P is converted into magnesium phosphide, which can be decomposed with dil. H_2SO_4 to give phosphine. This is absorbed in bromine water and oxidised to phosphoric acid, which can be determined photometrically as molybdophosphoric acid after having been extracted with ethyl acetate. The presence of N, S and halides does not cause interference. The method is suitable for 1·0 to 2·5 mg of sample, and the error is $\pm 0{\cdot}4\%$. The time required for one analysis does not exceed 100 min.

VI. Colorimetric micro-determination of antimony in organic compounds. J. Jeník. *Ibid.*, 1957, **51** (7), 1316–1319.—When organic compounds containing Sb are burned with metallic magnesium, the Sb is converted into magnesium antimonide, which can be decomposed with dil. H_2SO_4 to form stibine. This is absorbed in 6 *N* HCl containing alkali nitrite. The resulting hexachloroantimonic acid can be determined colorimetrically, after having been converted into a blue compound by interaction with methyl violet and extracted with toluene. The presence of N, halides, P and As does not cause interference. When analysing 1 to 1·5 mg of sample, the error is $> \pm 0{\cdot}4\%$. The time for a determination is 35 min. J. ZÝKA

856. **Gravimetric micro-determination of carbonyl groups by hydrazone formation.** T. S. Ma, J. Logun and P. P. Mazzella (Brooklyn Coll., New York, U.S.A.). *Microchem. J.*, 1957, **1** (1), 67–73.—The gravimetric method described is based on the condensation of the carbonyl compound with 2:4-dinitrophenylhydrazine to form an insoluble hydrazone. The reagent is a saturated soln. of the hydrazine in methanol containing oxalic acid (1%). The Benedetti-Pichler technique of three weighings is used with a reaction tube comprising a test-tube of 23 mm inside diam., 100 mm in length, and 35 ml in capacity. The Emich filter-stick is 120 mm long. *Procedure*—Dissolve the sample (0·05 to 0·15 millimole) in methanol and add the reagent soln. Remove the supernatant liquid and wash the ppt. free of the reagent. Dry at 75° and weigh to $\pm$ 0·01 mg. Aliphatic and aromatic aldehydes and ketones, and ketosteroids have been tested. The accuracy is within $\pm$ 0·3%. D. F. PHILLIPS

857. **Absorptiometric determination of methoxyl.** M. Langejan (Farm. Lab. Rijksuniv., Utrecht, Holland). *Pharm. Weekbl.*, 1957, **92** (19), 667–670.—The methoxyl group is converted by hydrolysis into methanol and the distilled methanol is oxidised to formaldehyde by $KMnO_4$ in H_3PO_4 at 0°, and determined absorptiometrically after condensation with chromotropic acid (*cf.* Mathers and Pro, *Anal. Abstr.*, 1956, **3**, 739). A modification of the method is described which enables it to be used on the micro scale. Several alkaloids were determined, and it was found that, in narcotine and narceine, the methylenedioxy group does not interfere. P. RENTENAAR

858. **Argentimetric method for epoxides.** G. A. Stenmark (Shell Development Co., Emeryville, Calif., U.S.A.). *Anal. Chem.*, 1957, **29** (9), 1367–1369.—The method is based on the reaction of the α-epoxy group with HCl in dioxan soln. followed by titration of the excess of Cl^- with $AgNO_3$, with $Fe(SCN)_3$ as indicator. Recoveries in the presence of amines were $> 99\%$. G. P. COOK

859. **Identification of organic compounds. XIX. Micro-identification of the lower aliphatic alcohols, O-alkyl and N-alkyl groups by paper chromatography.** M. Večeřa, J. Gasparič and A. Spěvák (Res. Inst. Org. Synth., Pardubice-Rybitví, Czechoslovakia). *Chem. Listy*, 1957, **51** (8), 1554–1556.—The sample is decomposed to give the alkyl halide, which is converted into the corresponding alkyl 3:5-dinitrobenzoate, and identified on the micro scale (15 to 60 μg) by paper chromatography, with the use of 1-naphthylamine (1% ethanolic soln.) as detecting agent. J. ZÝKA

860. **Microbiological method for micro-determination of ethanol and butanol in mixtures.** B. Rosenfeld (Charles H. Best Inst., Univ. of Toronto, Canada). *Annalen*, 1957, **607** (1–3), 144–150.—Washed suspensions of *Bacillus pasteurianum* convert butanol aerobically into butyric acid, and ethanol into CO_2 and H_2O. In the presence of 0·001 *M* arsenite, both alcohols are converted into the corresponding acids. All the reactions are stoicheiometric, and the use of these methods and the conventional Warburg technique enables 1 μmole of ethanol in admixture with 6 μmoles of butanol to be determined. The standard deviation of quadruplicate estimations is $\pm$ 0·1 to $\pm$ 0·2 μmole. A. G. COOPER

861. **Separation and determination of glycols.** R. N. Sargent (Rutgers Univ., New Brunswick, N.J., U.S.A.). *Dissert. Abstr.*, 1956, **16**, 1809–1810.—A method of separating mixtures of glycols by anion-exchange chromatography of their borate complexes, with procedures applicable to diethylene

glycol, ethanediol, propane-1:2-diol, glycerol, and the *meso*- and (±)-isomers of butane-2:3-diol, are described. S.C.I. ABSTR.

862. **Spectrophotometric method for the determination of periodate consumed during the oxidation of carbohydrates.** G. O. Aspinall and R. J. Ferrier (Univ., Edinburgh, Scotland). *Chem. & Ind.*, 1957, (36), 1216.—The oxidation of monosaccharides, hexitols and methylglycosides by periodate was followed by measuring the decrease in light absorption due to periodate and correcting for absorption due to iodate formed. Samples (6 to 9 mg of a monosaccharide consuming 2 moles of periodate, or 12 to 18 mg of a similar substance consuming 1 mole) were dissolved in 0·015 *M* $NaIO_4$ (10 ml) and kept in the dark at 35°. Aliquots were diluted 250-fold and the extinctions at 223 mμ were compared with those of periodate soln. and of an equimolar soln. of iodate. The reaction was complete in 8 hr. The reaction can be followed with 2 to 3 mg of carbohydrate by reducing the vol. Results are in good agreement with those obtained by the volumetric method.

G. BURGER

863. **Rapid method for the detection of reducing sugars on circular paper chromatograms.** H. Venner and W. Herb (Inst. für Mikrobiol. und Exp. Therapie, Jena). *Hoppe-Seyl. Z.*, 1957, **308** (1), 30–35.—Spots of sugar soln. containing 30 to 100 μg of sugar are chromatographed on filter-paper, with butanol - acetone - aq. NH_3 - water (40:50:3:15) as the developing solvent. Other solvent systems are described. Up to eight tests can be run on one paper. Deoxy sugars are detected with *p*-nitrophenylhydrazine, and other reducing sugars are located with aniline hydrogen phthalate or 4:4′-di-[2-(3 : 5-diphenyl-3*H*-tetrazolium-2-chloride)] stilbene. Details are given of the conversion of the carbohydrates into the heavy-metal complexes of formazan, and their detection as derivatives of tetrazotised diaminostilbenedisulphonic acid.

F. POWELL

864. **Rapid method for the quantitative estimation of reducing sugars on circular paper chromatograms.** H. Venner and W. Herb (Inst. für Mikrobiol. und Exp. Therapie, Jena). *Hoppe-Seyl. Z.*, 1957, **308** (1), 36–42.—For the estimation of reducing sugars 4 : 4′-di-[2-(3 : 5-diphenyl-3*H*-tetrazolium-2-chloride)]stilbene (**I**) is superior to 2:3:5-triphenyltetrazolium chloride. A weaker soln. of reagent can be used, the alkali need not be so strong, and as a result the paper blank is lower. The blank value can be lowered still further by impregnating the paper with H_3PO_4 or phosphate buffer. A further advantage is that the product of reaction is not sensitive to light. Equal vol. of 0·25% soln. of **I** and *N* methanolic NaOH are mixed just before use. The treated chromatogram is heated at 90° to 96° in a water-satd. atmosphere, when deep-blue spots are formed on a purple background. Washing in methanol removes the background, and excess of reagent is removed by washing with water. The spots are eluted with hot pyridine and the extinction is read at 586 mμ. The maximum deviation in a visual photometer is ± 3%; in a photo-electric instrument it is ± 1·3%. F. POWELL

865. **Ferricyanide and a modified periodate chromatographic spray for reducing and non-reducing sugars.** D. F. Mowery, jun. (Ripon Coll., Wis., U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1560–1561.—The addition of 25% *tert.*-butyl alcohol to an aq. soln. of sodium periodate reduces the spreading of spots on paper when the soln. is used as a spray for detecting reducing and/or non-reducing carbohydrates. If, after spraying the chromatogram with this soln., and drying at room temp., the paper is sprayed with a benzidine - NH_4NO_3 soln. in 50% *tert.*-butyl alcohol, white or yellow spots on a blue ground are produced with both reducing and non-reducing sugars. A spray of $K_3Fe(CN)_6$ and K_2PO_4 in 25% *tert.*-butyl alcohol, followed, after drying at 80° to 90° for 5 min., by spraying with the benzidine reagent, gives white spots on a blue ground with reducing sugars only. All the soln. are stable for 6 months.

R. E. ESSERY

866. **Evaluation of quantitative sugar analysis by paper chromatography.** R. H. Quick (Inst. of Paper Chem., Appleton, Wis., U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1439–1441.—In the chromatographic characterisation of hemicellulose hydrolysates, ethyl acetate - acetic acid - water (9:2:2) separates L-arabinose from D-mannose, and *n*-butanol - pyridine - water (10:3:3) separates D-glucose from D-galactose. These four, together with D-xylose and L-rhamnose, can be satisfactorily separated on a single paper by using the two solvent systems in succession in a specified manner, the sugars being eluted with water and determined by the periodate oxidation method. Recoveries from a known mixture of sugars ranged from 90 to 107%. The precision was better when the sugars were eluted completely from the paper into the reaction flask than when the paper was included with the reaction mixture. R. E. ESSERY

867. **Quantitative determination of lactose and monoses in lactose hydrolysates. Rapid horizontal paper-chromatographic method.** H. R. Roberts (Nat. Dairy Products Corp., Oakdale, Long Island, N.Y., U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1443–1447.—Apparatus is described and illustrated for horizontal paper chromatography at 60° with butanol - pyridine - water (9:5:8). Two developments, each of 1 hr., separate galactose and glucose (together) from lactose, and four such developments separate lactose from galactosyl oligosaccharides. The spots are developed by dipping the chromatogram into butanol containing benzidine and citric acid, the maximum densities are measured with a densitometer, and results are referred to a standard curve of log concn. *vs.* density. Comparison with a descending chromatographic method showed errors < 5%. R. E. ESSERY

868. **Formamide as an extraction solvent in Karl Fischer method for determining moisture in lactose and maltose.** E. A. McComb (W. Regional Res. Lab., Albany, Calif., U.S.A.). *Anal. Chem.*, 1957, **29** (9), 1375.—Formamide is a good extraction solvent for use in the Karl Fischer method, for determining both the water of crystallisation and adsorbed water in lactose and maltose. Results were in good agreement with those obtained by the direct oven-heating procedure. Formamide might readily be adapted for products containing these sugars. G. P. COOK

869. **Separation and quantitative determination of methyl mannosides in an automatically controlled cellulose partition column.** D. F. Mowery, jun. (Ripon Coll., Wis., U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1451–1453.—An analytical column (1 in. × 30 in.), packed with 130 g of cellulose powder,

and completely automatic, is described and illustrated. In the analysis of a mixture of mannose and 4 mannosides, with butanol - pyridine - water (10:3:3) and analysis of the fractions with anthrone reagent, the amount of a component found was usually within 2 units of that present over the range 1 to 57%. The working time for a complete analysis was ≃ 2 hr., and the chromatographic separation required ≃ 12 hr., at a rate of 1 ml per minute. R. E. ESSERY

870. **Micro-methylation of polysaccharides.** H. S. Isbell, H. L. Frush, B. H. Bruckner, G. N. Kowkabany and G. Wampler (Nat. Bureau of Standards, Washington, U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1523–1528.—An apparatus is described for methylation of a charge of 16 mg of polysaccharide, suitable for the production of labelled derivatives, the procedure being based on the liquid NH_3 macromethods of Muskat (*J. Amer. Chem. Soc.*, 1934, **56**, 693, 2449). Methylation takes place in a closed system, small measured amounts of alkali metal, contained in a thin glass capillary, being cut off in the bore of a stopcock, and washed into the reaction flask with liquid NH_3. Several procedures are described for the introduction of the methyl iodide. Diatomaceous earth in the reaction flask provides a large surface, and dimethoxyethane is used as a solvent for labelled methyl iodide. After methylation, the product is hydrolysed with formic acid in a sealed tube under CO_2, and after passage through a mixed-bed de-ioniser, the derivatives are chromatographed with ammoniacal butanol. R. E. ESSERY

871. **Flame-photometric determination of calcium in cellulose.** L. H. Phifer (American Viscose Corp., Marcus Hook, Pa., U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1528–1531.—Details are given of a H_2SO_4 - H_2O_2 wet-ashing process for cellulose. The residue is heated to fuming, then cooled and made up to standard vol. with water. The soln. is examined flame photometrically at 422·7 mμ and the concn. of Ca^{2+} obtained from standards. A HCl soln. obtained after ignition of the sample may also be used. No interferences are encountered. Precision data are tabulated for the range 8 to 500 p.p.m., and results are compared with those of the standard titrimetric oxalate method. D. A. PANTONY

872. **Regeneration of carbonyls from 2:4-dinitrophenylhydrazones with laevulic acid.** M. Keeney (Dairy Dept., Univ. of Maryland, College Park, U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1489–1491.—The regeneration is carried out by heating the hydrazone in a steam bath for 5 min. with 50 to 200 times (according to the solubility of the hydrazone) the weight of laevulic acid soln. If the parent substance has no unsaturation conjugated with the carbonyl group, the reagent is melted laevulic acid - water (9:1, by vol.), but when conjugated unsaturation is present, the laevulic acid is diluted with 1 vol. of *N* HCl or H_2SO_4. The regenerated substance can be recovered by steam-distillation or extraction, or, if odorous, recognised by smell directly or after dilution with water. The method has been used for the study of flavour compounds in dairy products after chromatographic separation of the hydrazones.
R. E. ESSERY

873. **Chromatographic separation of 2:4-dinitrophenylhydrazones.** E. L. Pippen, E. J. Eyring and M. Nonaka (U.S. Dept. Agric., Albany, Calif., U.S.A.). *Anal. Chem.*, 1957, **29** (9), 1305–1307.—The chromatographic behaviour of 2:4-dinitrophenylhydrazones of aliphatic aldehydes and ketones on silicic acid - celite columns was investigated. A column packed to a height of 75 cm permitted the separation of mixtures of hydrazones of saturated normal aldehydes up to C_{11}; beyond this point difficulty was encountered. Some chromatographic properties of 34 other 2:4-dinitrophenylhydrazone derivatives of aliphatic aldehydes are tabulated. G. P. COOK

874. **Analysis of formaldehyde and glyoxylic acid.** A. R. Ronzio (Radiological Res. Inst., Inc., Denver, Colorado, U.S.A.). *Microchem. J.*, 1957, **1** (1), 59–66.—A comparative study of three gravimetric methods and one volumetric method for the determination of formaldehyde was made. The gravimetric procedures were—(*i*) pptn. of the dimedone derivative; (*ii*) pptn. of the 4-hydroxycoumarin derivative (dicoumarol); (*iii*) oxidation with silver nitrate and triethylamine. The volumetric determination (*iv*) comprised addition of 25 ml of formaldehyde to about 40 ml of standard base (0·45 *N*) followed by oxidation with H_2O_2 and titration with standard acid. Of these, (*i*) and (*iv*) are recommended. It was found that the pptn. of dicoumarol is quant. when the compound is formed in the presence of an excess of 4-hydroxycoumarin, but not in the presence of an excess of formaldehyde. For the quant. determination of glyoxylic acid, oxidation with Tollens' reagent may be used. In the determination of glyoxylic acid by the pptn. of di-(4-hydroxycoumarinyl)acetic acid with 4-hydroxycoumarin, an excess of glyoxylic acid lowers the yield. D. F. PHILLIPS

875. **Determination of total hydrocyanic acid in acetone cyanohydrin.** Hiroo Sasuga (Inst. of Techn.-anal. Chem., Fac. of Engng, Tokyo Univ., Hongo, Tokyo). *J. Chem. Soc. Japan, Ind. Chem. Sect.*, 1957, **60** (4), 504.—The determination of CN in acetone cyanohydrin (**I**) by its decomposition with aq. NH_3 soln., followed by titration with $AgNO_3$, gives low values. This appears to be due to incomplete decomposition of **I**. The use of 10% NaOH (< 10 ml) is suggested. The sample soln. (2 to 5 g of **I** in 1 litre of water) (30 ml) is mixed with water (50 ml), 10% KI (1 ml) and 10% NaOH (5 ml) and titrated with 0·1 *N* $AgNO_3$ until a yellowish-green turbidity appears. K. SAITO

876. **Rapid procedure for separating C_2 to C_6 volatile fatty acids by horizontal paper chromatography at elevated temperatures.** H. R. Roberts and W. Bucek (Nat. Dairy Products Corp., Oakdale, Long Island, N.Y., U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1447–1449.—The acids are separated as their ethylamine salts in 1 hr. at 50°, with water-saturated *n*-butanol. Stable purple spots on a yellow background are developed by dipping the air-dried chromatogram in a soln. of chlorophenol red. If development is carried out for 2 hr., the spots are suitable for measurement by either the area or the max. spot-density method. If acids higher than C_6 are present, development at 60° gives better resolution between hexoic and heptoic acids. (See also Roberts, *Anal. Abstr.*, 1958, **5**, 867.) R. E. ESSERY

877. **Ion-exchange micro-methods for separation of fermentation acids. Determination of fumaric acid in fermentation broth.** C. H. Van Etten and C. E. McGrew (N. Utilization Res. and Devel. Div., Agric. Res. Service., Peoria, Ill., U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1506–1509.—The use of ion-exchange resins in micro columns with loads of

0·15 milli-equiv. of total acids is described. For the separation of fumaric acid, the fermentation liquor is passed first through a column of Dowex 50-X2 (H) resin and then through a column of Dowex 1-X1 (OH). The weak acids are eluted from the latter column with 0·35 *N* acetic acid, after which the strong ones are eluted with 0·1 *N* HCl. The latter eluate is evaporated to dryness, dissolved in 90% *tert.*-butyl alcohol and passed through a column of Dowex 1-X8 (OH), from which the fumaric acid is eluted with 0·35 *N* formic acid. The recovery of added fumaric acid was 98·2 ± 1·52%. R. E. ESSERY

878. **Spectrophotometric determination of microgram quantities of oxalic acid with copper-benzidine complex.** Z. D. Draganić and I. G. Draganić (Lab. of Phys. Chem., Inst. Nucl. Sciences, Belgrade). *Bull. Inst. Nucl. Sci., Belgrade*, **1957**, **7**, 53–58.—The following soln. were prepared. (*A*) Benzidine (32·5 mg) is dissolved in 2 ml of 30% acetic acid and diluted with water to 250 ml. (*B*) Copper acetate (125 mg) is dissolved in 250 ml of water. The reagent obtained by mixing *A* and *B* has a pH of 3·8, and gives with oxalic acid an absorption spectrum with max. at 248 mμ. The accuracy of the method is within 3% and Beer-Lambert's law is valid. The reaction of oxalic acid with the reagent is instantaneous at room temp., but is not specific, citric and tartaric acids interfering more seriously than formic and glycollic acids. Hydrogen peroxide does not interfere but foreign ions exert considerable influence on the absorption. J. H. W. FORSYTHE

879. **Specific spot tests for nitromethane and nitroethane based on the Nef degradation.** F. Feigl and D. Goldstein (Lab. Prod. Mineral, Min. Agric., Rio de Janeiro, Brazil). *Anal. Chem.*, **1957**, **29** (10), 1521–1522.—Strong mineral acid produces formaldehyde and acetaldehyde from nitromethane and nitroethane, respectively. For nitromethane, a drop of test material is shaken with a drop of 2% NaOH soln. After a few minutes, 3 to 4 drops of a fresh suspension of chromotropic acid in conc. H_2SO_4 are added and the mixture is warmed in a water bath. A violet colour indicates nitromethane (limit, 2·5 μg). For nitroethane, a drop of alkaline methanolic test soln. is treated with 3 drops of H_2SO_4 (1:1), care being taken not to wet the upper walls of the test-tube, the mouth of which is then covered with filter-paper wetted with a fresh mixture of equal vol. of 20% aq. piperidine (or morpholine) and 5% aq. sodium nitroprusside. On placing the tube in a boiling-water bath, a blue stain appears on the paper (limit, 8 μg). R. E. ESSERY

880. **Spot tests for nitromethane, monochloro-(bromo)acetic acid, dimethyl sulphate, iodomethane and methylsulphuric acid.** F. Feigl and D. Goldstein (Lab. Prod. Mineral, Min. Agric., Rio de Janeiro, Brazil). *Anal. Chem.*, 1957, **29** (10), 1522–1523.—In alkaline soln., nitromethane yields a blue-violet colour with 1:2-naphthaquinone-4-sulphonic acid in the cold. When CaO is used as the OH^- donor, the limit of detection is 0·6 μg. The other substances mentioned yield nitromethane on treatment with $NaNO_2$. Under the conditions given, the limits of detection (in μg) are—chloroacetic acid, 5; bromoacetic acid, 10; dimethyl sulphate, 50; iodomethane, 80; methyl hydrogen sulphate, 100. Ethyl hydrogen sulphate yields nitroethane, which does not react with the naphthaquinone. R. E. ESSERY

881. **Colorimetric determination of amino nitrogen in coloured solutions.** C. E. Weill and A. Bedekian (Rutgers Univ., Newark, N.J., U.S.A.). *Microchem. J.*, 1957, **1** (1), 89–91.—The method of Spies and Chambers (*J. Biol. Chem.*, 1951, **191**, 787), based on the formation of a soluble copper complex, is adapted to the determination of amino nitrogen in the range 0·03 to 0·4 millimole. The soln. of amino acid is treated with a suspension of copper phosphate in borate soln. After 1 hr. an aliquot containing from 10 to 25 μg of Cu as the complex is treated with Na diethyldithiocarbamate in the presence of citric acid-HCl soln. The coloured copper complex is extracted with amyl acetate-methanol, dried with anhyd. Na_2SO_4, and the extinction is measured. The calibration curve is prepared by measurement of dilutions of a standardised soln. of $CuCl_2$. As little as 0·003 millimole can be determined if it is not necessary to have results over 90% of the true value. D. F. PHILLIPS

882. **Colorimetric determination of primary amine in fatty amine acetates and fatty amines.** A. J. Milun (General Mills, Inc., Minneapolis, Minn., U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1502–1504.—The sample (≯ 1·6 millimoles) is dissolved in $CHCl_3$, then treated successively with $CHCl_3$ acidified with glacial acetic acid and with a soln. of salicylaldehyde in $CHCl_3$, and heated for 80 min. at 30°. A yellow Schiff's base is formed having an absorption max. at 410 mμ. Interference by secondary and tertiary amines is eliminated by the presence of acetic acid, and unsubstituted amides, N-alkylacetamide and nitrile do not interfere. The specified experimental details must be closely adhered to. Molar absorptivities of 8 amines and 1 amine acetate are tabulated. Recoveries of primary fatty amines alone and in the presence of the corresponding secondary and tertiary compounds were satisfactory, the 99% confidence interval for the mean being ±0·6%. The colour reaction may be used as a test for primary alkylamines. R. E. ESSERY

883. **Organic chemical microscopy. I. Amine picrates in qualitative organic analyses.** R. E. Dunbar and J. Knuteson (State Coll., Fargo, N. Dakota, U.S.A.). *Microchem. J.*, 1957, **1** (1), 17–37.—Crystalline products were obtained for 36 liquid and 14 solid amines by one or other of the following procedures. *Procedure A*—Add the amine (1 ml) to a hot saturated soln. of picric acid in ethanol (10 ml) (prepared at room temp. and then heated to b.p.). Allow the soln. to cool slowly and then filter off the crystalline picrate. Recrystallise the crude product twice, usually from 95% ethanol. If the amine used is a solid, first dissolve it in boiling ethanol and add its hot soln. to the boiling reagent. *Procedure B*—As for *A* except that the reagent is a benzene soln. of picric acid saturated at room temp. Dissolve solid amines in boiling benzene before mixing with the reagent. Recrystallise from water or aq. ethanol. Use *B* when *A* fails to give a derivative owing to the solubility of the picrate in ethanol. Of the 50 distinctly different picrate derivatives prepared, five, namely those of 2-amino-1-butanol, 3-aminodibenzofuran, *cyclo*hexamine, 2:5-dichloroaniline and ethylenediamine, are apparently reported for the first time. Significant data for these picrates are tabulated, and nitrogen determinations proved them all to be monopicrates. (52 photomicrographs.) D. F. PHILLIPS

884. **Determination of ammonium salts in acid mixtures containing guanidine salts.** R. W. Williams, H. Stalcup and M. I. Fauth (U.S. Naval Powder Factory, Indian Head, Md., U.S.A.). *Anal. Chem.*, 1957, **29** (9), 1356–1357.—The method is based on the reaction of ammonia with formaldehyde in a faintly acid medium, 1 equiv. of acid being liberated for each equiv. of ammonium salt. The determination can be carried out in the presence of nitric acid, guanidine salts and nitroguanidine. *Procedure*—The sample containing $\simeq 1$ g of ammonium salts is weighed into a 250-ml beaker suitable for potentiometric titrations, 100 ml of water is added and the mixture is stirred magnetically. The soln. is adjusted to $\simeq$pH 5 with 5 *N* NaOH and then to exactly pH 6 with 0·5 *N* NaOH. Formaldehyde soln. (35 to 40%) (15 ml), neutralised to pH 8·4, and 0·5 *N* NaOH are then added simultaneously so that the pH of the soln. in the beaker does not drop below 6, and titration is continued to an end-point at pH 8·4. G. S. ROBERTS

885. **Polarographic determination of xanthates in flotation liquors.** Shiou-Chuan Sun and R. T. Holzmann (Coll. of Mineral Ind., The State Univ., University Park, Pa., U.S.A.). *Anal. Chem.*, 1957, **29** (9), 1298–1300.—In this rapid polarographic determination of xanthate in water and mineral pulps, a supporting medium of NaOH - KCl is used with eosin added to extend the range down to 0·05 to 2·5 millimoles per litre. Ions that would normally interfere (*e.g.*, S^{2-}, CO_3^{2-}, SO_3^{2-} and SO_4^{2-}) are generally present in amounts too small to cause serious difficulty. K. A. PROCTOR

886. **Analysis of the system *cyclo*hexanone - *cyclo*hexanol by means of the dielectric constant.** D. Šnobl and L. Lehar (Res. Inst. Org. Synth., Pardubice-Rybitví, Czechoslovakia). *Chem. Průmysl*, 1957, **7** (8), 402–404.—A method for the analysis in the concn. range from 0 to 100%, the accuracy being $\pm$ 0·2%, is reported. The method is based on the measuring of the dielectric constant of the sample in benzene soln. J. BÖSWART

887. **Micro-colorimetric method for o-phthalates.** M. H. Swann (Aberdeen Proving Ground, Md., U.S.A.). *Anal. Chem.*, 1957, **29** (9), 1352–1353.—The method is based on the condensation reaction between phthalic acid or anhydride and quinol; the highly coloured compound quinizarin is formed. The yellow colour is extracted into benzene and the absorbance is measured at 480 mμ. Both maleic and succinic acids give coloured products with quinol, but neither interferes with the phthalate determination. Some typical alkyd resins gave results that were in reasonable agreement with those obtained by a u.v. procedure. The method has been applied to the determination of succinates in the absence of phthalic acid. G. P. COOK

888. **Chromatography of dyestuff intermediates. VII. Identification of the toluidines and nitrotoluenes by paper chromatography.** J. Latinák (Východočeské Chem. Závody, Pardubice-Rybitví, Czechoslovakia). *Chem. Listy*, 1957, **51** (8), 1493–1496.—The isomeric toluidines when converted into bromo derivatives can be chromatographically separated on paper impregnated with liquid paraffin, with the use of the system ethanol - acetic acid - water. The nitrotoluenes can be identified in the same manner, after reduction to toluidines. The method is suitable for the control of technical products. J. ZÝKA

889. **Identification of organic compounds. XVIII. Chromatography of aromatic hydrazo compounds.** M. Večeřa, J. Petránek and J. Gasparič (Res. Inst. Org. Synth, Pardubice-Rybitví, Czechoslovakia). *Chem. Listy*, 1957, **51** (8), 1553–1554.—A method is described for microgram amounts in which the solvent system was formamide - *cyclo*hexane, and *p*-dimethylaminobenzaldehyde in ethanolic HCl was used for detection. J. ZÝKA

890. **Colorimetric determination of certain organophosphorus compounds and acylating agents. Use of di*iso*nitrosoacetone reagent.** S. Sass, W. D. Ludemann, B. Witten, V. Fischer, A. J. Sisti and J. I. Miller (Army Chem. Center, Md., U.S.A.). *Anal. Chem.*, 1957, **29** (9), 1346–1349.—Dioximinoacetone reacts with organophosphorus halides, acid anhydrides and acylating agents to give an intense magenta colour, which can be measured either at 486 or 580 mμ. Sensitivities of 1 μg or lower can be obtained according to the final dilution of the colour-development medium. Recoveries of $> 97\%$ were obtained. G. P. COOK

891. **Partially deactivated silica gel columns in chromatography. Chromatographic behaviour of benzo[α]pyrene.** H. J. Cahnmann (Nat. Inst. Health, Bethesda, Md., U.S.A.). *Anal. Chem.*, 1957, **29** (9), 1307–1311.—Silica gel, partially activated by the addition of appropriate quantities of water, gave better defined chromatographic zones than a non-deactivated gel in the fractionation of many polycyclic aromatic hydrocarbons. Experiments with benzo[α]pyrene showed that, by the gradual addition of water to activated silica, an optimum degree of deactivation is reached, beyond which further deactivation produces poorer adsorbents. The R_F values of benzo[α]pyrene for the various activity grades of silica gel are presented and discussed. G. P. COOK

892. **Polarography of anthrone derivatives in glacial acetic acid.** L. Stárka and A. Vystrčil (Inst. Org. Chem., Charles' Univ., Prague, Czechoslovakia). *Chem. Listy*, 1957, **51** (8), 1449–1452.—Some derivatives of anthrone and dianthronyl yield, in a mixture of glacial acetic acid with 10% H_2SO_4, diffusion currents, due to their irreversible reduction on the dropping-mercury electrode. The polarographic waves are well developed and suitable for analytical purposes. J. ZÝKA

893. **Polarography of anthraquinone derivatives in glacial acetic acid.** L. Stárka, A. Vystrčil and B. Stárková (Inst. Org. Chem., Charles' Univ., Prague, Czechoslovakia). *Chem. Listy*, 1957, **51** (8), 1440–1448.—The polarographic behaviour of some substituted derivatives of anthraquinone and helianthrone is described. In a mixture of glacial acetic acid with 10% H_2SO_4 these compounds yield diffusion currents, accompanied with adsorption phenomena. The polarographic determination of anthraquinones and their distinction from anthrones is possible. The photo-dehydrogenation of helianthrone to naphthodianthrone was studied by a polarographic method. J. ZÝKA

894. **Spectra-structure correlations of alkylpyridines.** H. E. Podall (Purdue Univ., West Lafayette, Ind., U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1423–1426.—Infra-red spectra of alkylpyridines have characteristic absorption peaks in the region 11 to 15 μ, and characteristic band patterns in the 5 to 6-μ region, which depend on the position of

the alkyl groups in the pyridine ring, and also in the 7 to 9-μ region, which depend on the nature of the alkyl group. Ultra-violet absorption maxima can be correlated with substitution positions, but not with the nature of the alkyl group. Figures are given for pyridine and 29 alkyl derivatives. Infra-red correlations have been used for determining pyridine-base impurities in alkylpyridines and in following the course of purification procedures.
R. E. ESSERY

895. **Polarographic detection of 5-pyrimidinyl disulphides.** N. G. Luthy and B. Lamb (Chem. Dept., The Univ., Manchester, England). *Anal. Chem.*, 1957, **29** (10), 1454–1456.—The characteristic reduction-wave pattern of 5-pyrimidinyl disulphides in alkaline soln. is due to the –S·S– linkage, as shown by its disappearance on complete conversion to the thiol form. The main reduction wave at $-0{\cdot}58$ V *vs.* the S.C.E. is diffusion-controlled in the region 10^{-3} to 10^{-4} *M*. Other pyrimidine compounds containing a sulphur atom gave non-diffusion-controlled waves at much greater negative potentials. The reduction waves due to the disulphide linkage could be used for following the synthesis of 5-pyrimidinyl disulphide and for checking the homogeneity of fractions after recrystallisation. R. E. ESSERY

896. **Reductimetric determination of pyrazoleanthrone with the use of vanadium(II) sulphate.** M. Matrka and Z. Ságner (Res. Inst. Org. Synth., Pardubice-Rybitví, Czechoslovakia). *Chem. Prûmysl*, 1957, **7** (9), 484–485.—*Procedure*—Dissolve the sample (0·2 g) in ethanol (80 ml) and water (150 ml). Add a saturated soln. of $NaNO_3$ (25 ml) and titrate potentiometrically with 0·1 *N* VSO_4.
J. BÖSWART

897. **Determination of sulphoxides.** R. R. Legault and K. Groves (Dept. of Agric. Chem., State College of Washington, Pullman, U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1495–1496.—The method of Barnard and Hargrave (*cf. Brit. Abstr. C*, 1952, 145, 302) has been modified for application to sulphoxides which yield water-sol. sulphides on reduction with $TiCl_3$, and so give low and erratic results. In the present method, acetic acid is eliminated from the reduction step and interfering sulphides are removed by partial saturation with $(NH_4)_2SO_4$, and extraction with *n*-butanol followed by CCl_4, the final titration of the aq. phase with dichromate being carried out as in the original method. R. E. ESSERY

898. **Paraffin - cycloparaffin separations by liquid displacement partition chromatography.** R. W. Sauer, T. A. Washall and F. W. Melpolder (Atlantic Refining Co., Philadelphia, Pa., U.S.A.). *Anal. Chem.*, 1957, **29** (9), 1327–1331.—Paraffins and cycloparaffins in naphtha were separated by liquid partition chromatography with aniline as the stationary phase supported on silica gel. *iso*Propyl alcohol and benzene were used as desorbents and the sample fractions were displaced ahead of the desorbents so that the collected fractions were solvent-free. Refractive-index measurements were used to determine the composition of the test mixtures, and the hydrocarbon composition was obtained from mass-spectrometric analysis. The order of displacement from the column was paraffins, monocycloparaffins, dicycloparaffins and aromatics. Binary mixtures were successfully separated and fractions of purity of > 99% were recovered from naphtha. G. P. COOK

899. **Determination of physical properties on small samples of high-boiling hydrocarbons.** L. J. Sullivan, R. J. Fries, W. S. McClenahan and C. B. Willingham (Mellon Inst., Pittsburgh, Pa., U.S.A.). *Anal. Chem.*, 1957, **29** (9), 1333–1338.—Semi-micro methods are described for the determination of density and mol. wt. of hydrocarbon oils. Density is measured (*i*) by using a hydrostatic balance for samples as small as 0·5 ml, or (*ii*) by using a bi-capillary pyknometer for samples as small as 0·2 ml; the precision for both methods is within $\pm$ 0·001 g per ml. The mol. wt. determination is made by a boiling-point method with ebulliometers; as little as 5 ml of solvent is required. The average precision is within $\pm$ 2%. G. P. COOK

900. **A method for the structure analysis of mineral-oil fractions based on viscosity, refractive index and density.** J. Cornelissen and H. I. Waterman (Tech. Hogeschool, Delft). *Brennst-Chemie*, 1956, **37** (23–24), 404–408.—Graphs are presented by means of which that proportion of the total carbon which is contained in aromatic compounds (% C_A) and in naphthenic compounds (% C_N) can be obtained from a knowledge of the kinematic viscosity, sp. gr. and refractive index. These graphs are derived from a 3-dimensional diagram in which each set of values for the measured constants is represented by a point, and oil fractions having the same % C_A and % C_N values are represented by lines. Results obtained from these graphs are in satisfactory agreement with those obtained by the v - n - d and the n - d - M methods. Sulphur, if present, must be determined and a correction made to the C_N value. The advantage of the method lies in the direct use of the values log ν_{20}, n_D^{20} and d_4^{20} instead of complicated functions of these values. G. BURGER

901. **Determination of trace amounts of combined and elemental sulphur in petroleum fractions.** N. W Houghton [American Oil Co. (Texas), Texas City U.S.A.]. *Anal. Chem.*, 1957, **29** (10), 1513–1515.—The sample is burned in an oxy-hydrogen flame (details of burner and its operation are given) and the products of combustion are absorbed in dil. H_2O_2. This absorbing soln. is then treated with aq. NaCl and evaporated to low bulk, but not to dryness. The residue is made up to standard vol. with demineralised water and acidified with a little HCl. An aliquot is treated with solid $BaCl_2.2H_2O$ and, after swirling, the resulting suspension is measured turbidimetrically at 410 mμ with respect to a reagent blank. The concn. of S is derived from a calibration curve. Precision and accuracy data are tabulated, and, typically, a standard deviation of $\pm$ 0·6 to $\pm$ 1·0 p.p.m. in samples containing 2 to 11 p.p.m. of S is quoted, and a 99·2% recovery of S is claimed at the 50 p.p.m. level.
D. A. PANTONY

902. **Anodic polarographic estimation of aliphatic sulphides in petroleum.** H. V. Drushel and J. F. Miller (Mellon Inst. of Ind. Res., Pittsburgh, Pa., U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1456–1461.—Stationary platinum-wire electrodes are used with a special a.c. treatment before polarisation, and a mixture (7:3) of nitrobenzene and methanol, acidified with HCl, as solvent-electrolyte system. Experimental details have been described earlier (*cf. Anal. Chim. Acta*, 1956, **15**, 389). From a study of several dialkyl and alkyl aryl sulphides, a curve is obtained that shows the average effect of mol. wt. on the apparent diffusion coeff. By fractionating the crude sample into portions of narrow

mol. wt. range, the method can be used for the determination of aliphatic S. The precision attained by using the same electrode is $\simeq 3\%$, and with different electrodes is $\simeq 5\%$. R. E. ESSERY

903. **Determination of total sulphur in petroleum products. Combustion and disodium ethylenediaminetetra-acetate titrimetric method.** O. N. Hinsvark and F. J. O'Hara (The Girdler Co., Louisville, Ky., U.S.A.). *Anal. Chem.*, 1957, **29** (9), 1318–1322.—The sample is burned in an oxy-hydrogen flame and the products of combustion are collected in an H_2O_2 scrubber. The SO_4^{2-} formed in the scrubber are measured by adding an excess of Ba acetate and determining the excess of Ba^{2+} by EDTA titration, with Eriochrome black T as indicator. Samples containing from 100 to several thousand parts per million of S were successfully analysed without interference from halides or N. A max. standard deviation of $\pm$ 8 p.p.m. was obtained in the concn. range 100 to 200 p.p.m. and recoveries of added S to various petroleum samples were $> \simeq 97\%$. G. P. COOK

904. **Determination of sulphur in petroleum distillates with the thread-wick lamp.** W. R. Battles (Fletcher Oil Co., Wilmington, Calif., U.S.A.). *Anal. Chem.*, 1957, **29** (9), 1338–1346.—A CO_2 - O combustion procedure with a thread-wick lamp and a new design of chimney absorber gives a precision within 2 μg of S in the range 0 to 100 p.p.m. of S. As little as 2 ml of sample is sufficient for several determinations, even at concn. of S $<$ 10 p.p.m. The method was successfully applied to a large no. of distillates and blends which included thiophen - *iso*octane blends, butanethiol blends and diesel oil. Recoveries of S were generally much greater than 80%, and results were in good agreement with those obtained by methods with the ASTM lamp. The $BaCl_2$ turbidity procedure was sufficiently accurate, for the final sulphate determination, for contents of S $<$ 0·05%, but for higher concn. the gravimetric sulphate procedure is recommended. G. P. COOK

905. **Titrimetric determination of dissolved sulphur in mineral oils and greases.** F. Stinsky (i. Fa. Goldgrabe & Scheft, Bremen, Contrecarpe 18). *Erdöl u. Kohle*, 1957, **10** (4), 231.—The sample containing 5 to 80 mg of S is heated under reflux for 30 min. with 100 ml of acetone. Water (20 ml) and 1·5 ml of indicator soln. (0·04% of bromocresol purple in ethanol) are added and the liquid is titrated with 0·05 *N* KCN (3·25 g of KCN dissolved in 200 ml of water and made up to 1 litre with *iso*propyl alcohol) until the red - violet colour persists on further refluxing. The 0·05 *N* KCN is standardised against a soln. of S in acetone. Determinations on known amounts of S were correct to within 0·3%. Thiols interfere, but not disulphides or phosphoric esters. A. R. PEARSON

906. **Determination of tetraethyl-lead in gasoline.** J. J. Russ and W. Reeder (Campbell Taggart Res. Corp., Dallas, Tex., U.S.A.). *Anal. Chem.*, 1957, **29** (9), 1331–1332.—The tetraethyl-lead is extracted into conc. HCl and the Pb is pptd. as $PbSO_4$. The Pb is then determined by complexometric titration with EDTA (disodium salt) to Eriochrome black T indicator. There is no interference from Fe. The standard deviation for duplicate determinations is $\pm$ 0·002 for a range of 1·7 to 3·0 ml of tetraethyl-lead per gallon, and results were in close agreement with those obtained by the ASTM lead chromate procedure. G. P. COOK

907. **Determination of nitrogen in solid fuels by the Kjeldahl method. Study of catalysts and other variables.** F. González-Sánchez and V. Gómez Aranda (Inst. Nacional del Combustible, Zaragoza, Spain). *Combustibles*, 1957, **17**, 176–187.—The effect of rate of heating, temp., type and amount of catalyst and after-boiling period on the determination of N in low-grade coals has been examined. Rapid heating reduced both the clearing time and the after-boiling times required. The temp. of digestion depended on the amount of K_2SO_4 added and on the ratio of K_2SO_4 to H_2SO_4, but for normal additions was approx. 240°. Under specified conditions the relative orders of efficiency of the catalysts were Se - $HgSO_4$ (1 g), Se (0·25 g) and $HgSO_4$ (1 g), the digestion periods required ranging from 2 to 3 hr. L. A. O'NEILL

908. **Determination of nitrogen in coal by the Kjeldahl method.** Inst. Nac. del Combustible (Zaragoza, Spain). *Combustibles*, 1957, **17**, 188–189.—The procedure recommended is based on the investigations of González-Sánchez and Gómez Aranda (*cf. Anal. Abstr.*, 1958, **5**, 907). The powdered sample (1 g) is heated rapidly with K_2SO_4 (10 g), conc. H_2SO_4 (30 ml) and catalyst, which is optionally Se (0·25 g), $HgSO_4$ (1 g) or mixed Se and $HgSO_4$ (1 g). After clarification, the mixture is heated for 2 to 2·5 hr., cooled, treated with H_2O, alkali and Zn, and the NH_3 is distilled over into 0·1 *N* H_2SO_4 and titrated in the usual way. L. A. O'NEILL

909. **Chromatographic analysis of tars.** C. F. Cullis, J. A. W. Dalziel and J. E. Manton (Imperial Coll., London). *Chem. & Ind.*, 1957, (36), 1212–1214.—The constituents of tar obtained by the high-temp. pyrolysis of chlorobenzene vapour were separated by elution chromatography on an alumina column. The following eluents were used, in the order given—light petroleum, b.p. $<$ 40° (200 ml); 10% (v/v) ether in light petroleum (300 ml); 25% ether in light petroleum (200 ml); ether (400 ml); and ethanol (150 ml). The only pure compound isolated was *pp'*-dichlorodiphenyl. G. BURGER

910. **Rapid determination of carbon and hydrogen in tar, pitch and oils with high boiling-points.** A. R. Panicker and N. G. Banerjee (Central Fuel Res. Inst., Jealgora). *J. Instn Chem. India*, 1957, **29** (3), 145–149.—The method described is suitable for use with tar, anthracene oil (boiling range 270° to 300°), fuel oils (boiling range 210° to 360°), petroleum oils, and pitch. Liebig's procedure is modified by the introduction of a T-piece and a rod to push the boat with the sample to the desired hot zones. The rate of flow of O is 250 to 300 cu. cm per min. Evaporation of the oil sample is controlled by raising its temp. from 100° below its b.p. to a few degrees below its b.p. during 20 min. The method takes about 45 min. whereas Liebig's method takes about 4 to 5 hr. In repeated determinations the values for C and H agree to within 0·1 and 0·05%, respectively. O. M. WHITTON

911. **Rapid determination of ash in lignites.** F. González-Sánchez and R. Isábal Gracia (Inst. Nac. del Combustible, Zaragoza). *Combustibles*, 1957, **17**, 3–11.—An apparatus due to Pozzeto, in which coal samples are burnt in a current of O, is described. The apparatus is preheated and volatile material is driven off at high temp., avoiding inflammation. The carbon is then burnt off in a reduced current of gas and at lower temperature. Ten to twelve determinations can be made in 1 hr. The standard

error of the mean of duplicate determinations is ± 0·17%; the method gives lower results with certain Spanish lignites high in sulphur.

G. F. PENNY

912. **Determination of neutral oils and pyridine bases in cresylic acid.** G. E. Mapstone, A. T. Keppie and J. S. McFarland (South African Torbanite Mining and Refining Co. Ltd., Boksburg North). *S. Afr. Ind. Chem.*, 1957, **11** (3), 55–57.—Some improvements in the Standardisation of Tar Products Test Committee Method No. CC–5–55 are reported in detail. S.C.I. ABSTR.

913. **Gas chromatography and the perfumer.** W. A. Wiseman. *Perfum. Essent. Oil Rec.*, 1957, **48** (8), 380–385.—The technique and limitations of gas chromatography, as applied to the analysis of essential oils and perfumes, is described. Several chromatograms are illustrated and discussed.

H. B. HEATH

914. **Determination of camphor in Ho-leaf oil.** Teikichi Hiraizumi and Akira Komatsu (Takasago Perfumery Co. Ltd., Kamata, Tokyo). *Perfum. Essent. Oil Rec.*, 1957, **48** (8), 387–388.—The oxime method for the determination of camphor in Ho-leaf oil gives higher results than those obtained by i.r. analysis, owing to the presence of other carbonyl compounds. Ho-leaf oil is a two-component system of linalol and camphor, and its absorbance at 1743 cm^{-1} is used for the determination, the camphor content being obtained from a calibration curve, within the range of 0·5 to 2·6% of camphor. Ho-leaf oil contains approx. 1% of camphor.

H. B. HEATH

915. **Chromatography of waste sulphite liquor.** A. C. Shaw and M. Dignam (Dept. of Chem., Ontario Res. Foundation, Toronto). *Canad. J. Chem.*, 1957, **35** (4), 322–332.—A waste sulphite liquor from a soft cook of mixed spruce and balsam, and commercially fermented with a strain of *Saccharomyces cerevisiae*, was submitted to batch dialysis against dist. water through Cellophane membranes for 10 days. The dialysate, concentrated to 50% of solids, was then subjected to a multi-stage fractional pptn. with ethanol, and the sol. fraction was selected for further investigation. By a combination of chromatographic and ion-exchange techniques this was shown to contain a series of phenolic compounds which appear to be ligninsulphonate fragments, together with xylose, arabinose, galactose, rhamnose and polysaccharides.

A. JOBLING

916. **Washed flock (loose or layered).** British Standards Institution (2 Park St., London). B.S. 2894:1957, 20 pp.—This standard brings together the requirements for washed flock to be used as a filling material for bedding, and incorporates B.S. 1425 and B.S. 1877. Methods of sampling and for the determination of animal fibre, soluble chlorides, oil and soap, and extraneous matter, and a test for cleanliness are included.

N. E.

917. **Quantitative determination of ε-caprolactam in polymers by the Kjeldahl method.** P. Čefelín and E. Sittler (Inst. Technol. Plastics, High-School Chem. Technol., Prague, Czechoslovakia). *Chem. Listy*, 1957, **51** (7), 1320–1322.—A comparison of the determination of ε-caprolactam in aq. soln. and in polymers, gravimetrically and with the Kjeldahl method, has been made. The Kjeldahl method has been found to be more accurate. A mixture of ammonium metavanadate with potassium sulphate is the only suitable catalyst. J. ZÝKA

918. **Determination of foots in raw linseed oil.** H. Fähnrich (Dtsch. Linoleum-werke Akt., Delmenhorst, Germany). *Farbe u. Lack*, 1957, **63**, 5–12, 65–70.—The foots content of several samples of linseed oil as determined by various standard procedures and variations thereof have been compared. It is concluded that the max. information without the use of expensive apparatus can be obtained by carrying out three determinations—(*i*) the foots content by the B.S. method (a simple sedimentation at 20° for four days); (*ii*) the foots content by the B.S. procedure, but at a temp. of 40°; this gives by difference a measure of the amount of foots soluble in warm oil; (*iii*) the latent foots content on the clear oil layer by the Dutch standard method (treatment of the oil in acetone soln. with saturated aq. $CaCl_2$ containing HCl). L. A. O'NEILL

919. **Foots in linseed oil.** H. A. Boekenoogen (Vlaardingen, Holland). *Verfkroniek*, 1957, **30** (3), 53–54.—Procedures for determining the foots in linseed oil are critically reviewed and the nature of the phospholipids constituting the foots is considered. Raw linseed oil may contain up to about 0·12% of ash and 0·04% of P, which will be equivalent to ≃ 1% of phospholipids. L. A. O'NEILL

920. **Analysis of driers.** S. S. Manelkar (Bombay Univ.). *Paintindia*, 1957, **7** (1), 94–95.—In the complete analysis of liquid driers, the solvent is determined by heating at 105° and the metals by standard methods. Free acidity is determined by titration with alkali after treatment with alcoholic oxalic acid. The acid radical of the driers is examined after saponification of the drier and extraction with ether, the neutralisation equiv. giving an indication of the presence of naphthenic or fatty acids. L. A. O'NEILL

921. **Determination of polyhydric alcohols in paint media.** C. P. A. Kappelmeier and J. Mostert (Kunstharsfabriek Synthese N.V., Katwijk aan Zee, Holland). *Verfkroniek*, 1957, **30** (3), 48.—Polyhydric alcohol components of alkyd resins are separated by heating the resin with 2-phenylethylamine under reflux and then adding $CHCl_3$. Pentaerythritol crystallises immediately in the soln., followed, on cooling the soln., by trimethylolethane; the two alcohols cannot be quant. separated by this method, but their combined total may be found and the individual components estimated from the hydroxyl value. The filtrate will contain trimethylolpropane and glycerol, and the glycerol may be determined by reaction with KIO_4. Precautions to be taken with certain mixtures, *e.g.*, of glycerol and pentaerythritol, are indicated.

L. A. O'NEILL

922. **Determination of "hard resins" in paint media.** R. Dooper and J. A. M. van der Valk (Verfinst. T.N.O., Ryswijk, S. Holland). *Verfkroniek*, 1957, **30** (1), 10–11.—The proportion of the "hard" or "brittle" resins (such as rosin, ester gum, phenolic, coumarone, petroleum and *cyclo*hexanone resins, copal esters and run copal) in varnish-type paint media is taken as the total weight of unsaponifiable matter plus resin acids as determined by a slight modification of Kappelmeier's method (*Verfkroniek*, 1943, **16**, 68), the conditions of saponification and separation being such as to remove oils and alkyd resins (which are not included in the category of "hard" resins). The method is only approximate. D. R. DUNCAN

923. **Determination of phthalic anhydride in alkyd resin paints.** L. J. David (S.N.C.F. Lab., France). *Peint.-Pigm.-Vern.*, 1957, **33** (4), 331–332.—The medium is separated from the pigment by extraction with ether, the sap. val. is determined, and the phthalic anhydride is determined by a standard Kappelmeier procedure as K phthalate, after treatment with a calculated excess of KOH in absolute ethanol. L. A. O'NEILL

924. **Direct colorimetric determination of nitrocellulose in lacquers.** M. H. Swann (Coating and Chem. Lab., Aberdeen Proving Ground, Md., U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1504–1505.—An aliquot of an acetone soln. of the lacquer is refluxed for 1 hr. with an equal vol. of 10% aq. KOH soln., cooled, and made up to a known bulk with acetone - water (2:1). The yellow colour is measured at 425 mμ, and results are referred to a standard curve, which is rectilinear up to 45 mg of nitrocellulose. Interference is found with some rosin products and phenol - aldehyde condensates, which should be tested for before applying the method. Interference from aldehydes is eliminated by drying the sample aliquot at 105°.
R. E. ESSERY

925. **Determination of melamine - formaldehyde resins in coatings.** M. H. Swann and G. G. Esposito (Aberdeen Proving Ground, Md., U.S.A.). *Anal. Chem.*, 1957, **29** (9), 1361–1362.—The method is based on the insolubility of acid-hydrolysed melamine resins in dioxan, which is a good solvent for coating resins in general; the resins are determined gravimetrically. Urea resins, which may appear separately or in mixture with the melamine resins, also give quant. yields, but may be corrected for by application of a specific method for urea. Results obtained were in reasonable agreement with those by a u.v. procedure and a total nitrogen method, and recoveries were > 97%.
G. P. COOK

926. **Determination of cellulose resins in coatings by colorimetric cellulose analysis.** M. H. Swann (Coating and Chem. Lab., Aberdeen Proving Ground, Md., U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1505–1506.—Cellulose ethers and esters are determined by means of the blue - green colour developed when they are heated with H_2SO_4 (3:1) and anthrone at 90° for 20 min. Absorbancies are read at 625 mμ and, after corrections for blanks, are referred to a standard curve prepared from a known cellulose resin or pure cellulose. The colour is due to the cellulose moiety of the molecule and, for a resin to be determined, its nature must be known. Nitrocellulose interferes, but other resins and plasticisers do not. With control of experimental conditions, results are highly reproducible, and the colour is stable for 24 hr. R. E. ESSERY

927. **Estimation of sulphite-cellulose extract in syntans.** M. P. Balfe (Brit. Leath. Mfrs.' Res. Ass., Milton Park, Egham, Surrey). *J. Soc. Leath. Tr. Chem.*, 1957, **41** (4), 118–119.—The cinchonidine precipitation method for assessing the tanning agent lignin in sulphite-cellulose extract is not applicable when syntans are present. Syntans and lignin differ in that the former has little or no methoxyl content whereas the latter contains 12 to 20%. An approx. estimation of the lignin content in preparations containing syntans is obtained by determining the methoxyl content by Clark's method and multiplying by seven. G. HELMS

928. **Reduction of nitroglycerin by catalytic hydrogenolysis in analysis of propellents for dioctyl phthalate.** H. Stalcup, M. I. Fauth, J. O. Watts and R. W. Williams (U.S. Naval Powder Factory, Indian Head, Md., U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1482–1485.—Spectrophotometric, gravimetric and polarographic methods for the phthalate determination are given. Interference by nitroglycerin is eliminated by catalytic hydrogenolysis with palladium black or platinum oxide. For the spectrophotometric method, the propellent is extracted with 95% ethanol, and interference by 2-nitrodiphenylamine is removed by catalytic reduction and passage through a column of Amberlite IR-120. The phthalate is measured at 276 mμ, a measurement on the unreduced extract at 425 mμ permitting the estimation of the diphenylamine. When the diphenylamine is not required, the extractant is a pentane - dichloromethane azeotrope (2:1). For the spectrophotometric and gravimetric methods, the phthalate is determined as the dipotassium salt after saponification, and the polarographic determination is made directly on the filtered extract after hydrogenolysis.
R. E. ESSERY

929. **Determination of phthalate esters in propellents.** H. Stalcup, F. McCollum and C. L. Whitman (U.S. Naval Powder Factory, Indian Head, Md., U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1479–1482.—The phthalate is extracted from the propellent with dichloromethane, and the extract is treated with Zn and acetic acid to reduce nitrate esters and nitro compounds. A second extraction eliminates Zn and other insol. compounds, and the extract is saponified and the phthalic acid determined gravimetrically as the dipotassium salt or by titration with $HClO_4$ in glacial acetic acid soln. Recoveries were >99% except with dimethyl phthalate, when losses were 5 to 6%. R. E. ESSERY

See also Abstracts 766, 1048, 1080, 1081.

4.—BIOCHEMISTRY
INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

Blood, Bile, Urine, etc.

930. **New simple method for determining the oxygen content of blood.** A. G. Dembo and S. B. Tikhvinskiĭ (Sektor Vrach. Kontrolya, Leningr. N.-Iss. Inst. Fiz. Kult.). *Lab. Delo*, 1957, (3), 3–8.—Improvements are made in Scholander and Roughton's micro-method for determining O in blood (*J. Ind. Hyg. and Toxicol.*, 1942, **24**, 218). Manipulations are made with a 1 to 2-ml syringe, connected with a microcapillary 7 to 8 cm long, graduated in 0·001 ml, in which measurements of the vol. of gas are carried out; to the microcapillary is sealed a small funnel. Fill the syringe with a ferricyanide mixture [12·5 g of $K_3Fe(CN)_6$, 3 g of $KHCO_3$ and 0·5 g of saponin, made up to 50 ml]. Introduce a little capryl or octyl alcohol, then 0·1 ml of the blood, taking care not to let air into the apparatus. Introduce acetate buffer soln. (35 g of hydrated Na acetate in 50 ml of water and 15 ml of glacial acetic acid), then a little of a 45% soln. of urea. Close the funnel with a finger and shake to obtain gaseous O, N and CO_2. Absorb the CO_2 by drawing in 10% NaOH soln. from a

rubber cup. Remove the NaOH soln., stand the apparatus in water at room temp. for 30 sec. and measure the vol. of residual gas (O and N) in μl. Absorb O with alkaline pyrogallol soln. and measure the vol. of N. The results are reproducible to within ± 5% and agree with Van Slyke's method of determination within the same limits.

C. D. KOPKIN

931. **Some changes in the syringe method of determining the saturation of blood with oxygen.** V. A. Kropachev (Tsentral. Inst. Usover. Vrach., Klin. Bol'n. im. S.P. Botkin). *Lab. Delo*, 1957, (3), 9–11.—Improvements are made on Scholander's micro-method for determining O in blood. The microcapillary is 40 cm long, with 250 divisions of 0·0002 ml each, and may be read to 0·1 μl. The funnel is larger than in Scholander's instrument. The same soln. are used as in Scholander's method, except that a 5% soln. of $K_3Fe(CN)_6$ is used instead of a 25% soln. The greater resistance of the microcapillary eliminates any manual manipulation of the piston during the determination. The results of the method on 0·1 ml of blood are compared with those by Van Slyke's method on 1 ml, and showed discrepancies of $< \pm 2{\cdot}5\%$. C. D. KOPKIN

932. **Rapid spectrophotometric determination of the percentage of oxygenation and of the oxygen content of dogs' blood and comparison with the manometric method.** J. Carlier (Inst. de Clinique et de Polyclinique Médicales, Univ. de Liége, Belgique). *Bull. Soc. Chim. Biol.*, 1957, **39** (7–8), 995–1004.—The method described, previously applied to human blood (*cf. Acta Clin. Belg.*, 1956, **11**, 332), depends on absorption measurements in the red and i.r. regions. The difference in results by the spectrophotometric and Van Slyke methods ranged from −1·6 to +1·3% with a mean of − 0·3% and a standard deviation of ± 0·8%.

R. E. ESSERY

933. **Accurate flame-photometric estimation of sodium, potassium and calcium in urine.** A. Hilgers (Med. Abt. des Silikose-Forschinst. der Bergbauberufsgenossenschaft, Bochum, Germany). *Hoppe-Seyl. Z.*, 1956, **304**, 193–199.—Because the concentration of the various cations in urine varies over a range which is many times greater than the variation found in serum, the effect of the other two cations on the determination of the third is much more important and, unless due allowance is made, errors of ±10% may occur. With the procedures described it is claimed that the error is not more than ± 1%. A Zeiss flame photometer, model III, is used with air at 0·48 atm. pressure and acetylene at 80 mm (olive oil). F. POWELL

934. **Method for routine determination of sulphur-35 in biological material.** S. S. Walkenstein and C. M. Knebel (Wyeth Inst. for Med. Res., Radnor, Pa., U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1516–1517.—The method described is applicable to the simultaneous analysis of large numbers of samples and avoids the danger, inherent in sintered-glass filters, of radioactive contamination of successive samples. The complete analysis is carried out in a tared centrifuge tube (40 ml) modified to allow for easy packing and washing of the $BaSO_4$ ppt. The $BaSO_4$ is easily transferred to a planchet for counting, and the results are comparable in reproducibility to those obtained by filtration. K. A. PROCTOR

935. **Spectrophotometric determination of submicrogram amounts of nickel in human blood.** M. L. Cluett and J. H. Yoe (Univ. of Virginia, Charlottesville, U.S.A.). *Anal. Chem.*, 1957, **29** (9), 1265–1269.—The sample is ashed with HNO_3 and the mineral constituents are converted into chlorides with HCl. The Ni is complexed with Na diethyldithiocarbamate and extracted into *iso*amyl alcohol, and the extinction is measured at 325 mμ. Interference is caused by Fe^{III} and Cu^{II}, but these metals can be separated by anion exchange; Pb also interferes and is separated by adsorption on a column of $CaCO_3$. The coeff. of variation for 10 synthetic samples was 1% at the 0·5-μg level. Recoveries of added Ni were between ≃ 80 and 120% at the 0·05 and 0·1 p.p.m. levels. The concn. of Ni in blood was between 0·025 and 0·067 p.p.m.

G. P. COOK

936. **Quantitative determination of bone minerals from roentgenograms.** P. Virtama (Dept. of Anatomy, Univ. of Helsinki, Finland). *Experientia*, 1957, **13** (6), 236–237.—By this method the accuracy of bone-mineral determination is increased by avoiding the error due to uneven distribution. The images of the bones on the roentgenograms are cut out, their areas measured, and the silver contents determined by the indirect Volhard titration. The error is calculated to be ≃ 3·6%. P. NICHOLLS

937. **Improvements in or relating to glucose indicator.** Eli Lilly and Co. [Inventor: A. S. Keston]. Brit. Pat. 784,548; date appl. 7.7.55.—A test for glucose in liquids is based on the reaction of glucose in the presence of a glucose enzyme to give products that react with an indicating substance to yield a colour change.

H. F. W. KIRKPATRICK

938. **Spectrophotometric micro-determination of alcohol in blood.** D. Monnier, W. F. Rüedi and M. Fasel (Lab. de Chim. Minérale, Univ. of Geneva, Switzerland). *Mitt. Lebensmitt. Hyg., Bern*, 1957, **48** (3), 146–151.—The reagent for oxidation of the alcohol is prepared by dissolving 0·277 g of $K_2Cr_2O_7$ in 100 ml of H_2O, adding 94 ml of pure HNO_3 and diluting with H_2O to 250 ml. The apparatus is of the diffusion-unit type, capillary blood (0·1 ml), H_2O (0·5 ml) and saturated aq. K_2CO_3 (0·5 ml) being placed in the outer chamber, and the reagent (0·5, 1·0 or 1·5 ml according to the alcohol content) in the centre. After incubating at 50° for 2·5 to 3 hr. the colour of the reagent diluted 1 to 20 is read at 366 mμ. A blank and a standard are run with each test. H. F. W. KIRKPATRICK

939. **Photometric determination of haemoglobin.** V. V. Popov and B. A. Sobchuk (L'vovsk. Med. Inst.). *Lab. Delo*, 1957, (3), 19–20.—The extinction at 430 mμ of haemin chloride, obtained by dissolving blood in dil. HCl soln., is measured.

C. D. KOPKIN

940. **Comparative investigation of modern quantitative methods of estimating plasma fibrinogen.** A. Hirsch and C. Cattaneo (Ist. "C. Forlanini" dell' I.N.P.S. Clinica Tisiologica, Univ. Roma). *Hoppe-Seyl. Z.*, 1956, **304**, 53–64.—An investigation of the various methods was carried out which did not include the refractometric, dilution, heat-coagulation and viscosity methods, which were considered to be less accurate. On the whole, agreement between the results obtained by different techniques was good. Those methods that involve previous separation of the fibrin clot were found to show greater variation than those which estimate

the fibrinogen as such. Details are given of the technique regarded as most suitable from each group, *viz*, biuret determination of the fibrin ppt., ninhydrin estimation of amino nitrogen after alkaline hydrolysis of fibrin, nephelometric determination of fibrinogen after salting out with ammonium sulphate, and determination by paper electrophoresis. F. POWELL

941. **The estimation of iminazolylacetic acid in urine and its occurrence in urticaria pigmentosa.** A. Hanson (Chem. Zentrallab., Allg. Krankenhaus, Malmö, Sweden). *Naturwissenschaften*, 1957, **44** (17), 470–471.—Urine is adjusted to pH 8 and passed through a column of prepared Dowex-1X10 resin; elution is then carried out with *N* acetic acid and the eluate is evaporated to dryness under reduced pressure. The residue is dissolved in a little dil. acetic acid and chromatographed on paper in *n*-butanol - acetic acid - H_2O (4:1:5). The iminazolylacetic acid (R_F 0·33) is located by spraying with a 0·5% soln. of *p*-diazobenzenesulphonic acid in 5% Na_2CO_3 soln.; the coloured region is cut out and the colour is extracted with *tert.*-butyl alcohol - H_2O - 10% aq. Na_2CO_3 soln. (25:25:5) and read at 500 mμ. H. F. W. KIRKPATRICK

942. **Quantitative determination of histamine in presence of certain interfering metal ions.** A. C. Andrews and T. D. Lyons (Kansas State Coll., Manhattan, U.S.A.). *Anal. Chem.*, 1957, **29** (9), 1325–1327.—In the colorimetric procedure for histamine, in which the *p*-sulphonic acid diazonium ion is used as the coupling reagent, certain ions, such as Co^{2+}, Cu^{+}, Cu^{2+}, Ni^{2+} and Fe^{3+}, interfere by forming stable complexes with the histamine. These interferences are eliminated by the use of diethylenetriaminepenta-acetic acid (**I**), which forms stronger complexes with these ions than with the histamine. The stability constants for the **I** complexes are of the order of 10^{20}, whereas those of the histamine complexes are $\gtrsim 10^{16}$. G. P. COOK

943. **The separation of riboflavine derivatives by counter-current extraction.** F. Bergel, R. C. Bray and K. R. Harrap (Chester Beatty Res. Inst., Royal Cancer Hosp., London). *Annalen*, 1957, **607** (1–3), 219–224.—Flavins are separated by counter-current extraction by the use of a phenol - cresol - phosphate buffer system. The method is applied to the purification of commercially-available flavin mononucleotide (**I**), to the separation of **I** and flavinadenine dinucleotide (**II**), and to the isolation and characterisation of **II** in beef xanthine oxidase. A. G. COOPER

944. **Chromatographic detection of furan-2:5-dicarboxylic acid in human urine.** B. Flaschenträger and B. S. Kamel (Inst. for Biochem., Univ. Alexandria, Egypt). *Annalen*, 1957, **607** (1–3), 150–152.—Furan-2:5-dicarboxylic acid (**I**) has previously been shown to be present in urine by a tedious process of isolation (*cf.* Flaschenträger and Bernhard, *Hoppe-Seyl. Z.*, 1937, **246**, 126). In the chromatographic procedure now described, a conc. ethereal extract of acidified urine is treated with *n*-butanol - acetic acid - water (4:1:5), and **I** is detected by its colour reaction with an aq. soln. of aniline and xylose. A. G. COOPER

945. **Colour reaction of anthrone with monosaccharide mixtures and oligo- and poly-saccharides containing hexuronic acids.** J. R. Helbert and K. D. Brown (Marquette Univ. School of Med., Milwaukee, Wis., U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1464–1466.—The red colour produced by anthrone in 27·5 *N* H_2SO_4 with uronic acids has been used to resolve synthetic mixtures of galacturonic acid, galactose and rhamnose, by operating under three sets of experimental conditions and solving three equations for three unknowns. Beer's law is obeyed up to 200 μg per 6 ml at 540 and 625 mμ. The largest observed deviation from the known values was 10%, the major part of which is ascribed to variations in the preparation and manipulation of solutions. Conditions suitable for estimating monogalacturonic acid are also suitable for the di-, tri- and tetra-acids. Sodium heparinate gives the uronic acid colour with anthrone, but the intensity is greater than would be expected from the proportion of uronic acid present, the enhancement not being due to glucosamine. R. E. ESSERY

946. **The occurrence of glucuronic acid and fucose in human milk. Application to the quantitative determination of glucuronic acid by means of the 1:3-dihydroxynaphthalene reaction.** K. Heyns, W. Walter and W. Hyde (Chem. Inst., Univ. Hamburg). *Hoppe-Seyl. Z.*, 1956, **304**, 279–285.—When the 1:3-dihydroxynaphthalene reaction is applied to human milk it gives an "apparent glucuronic acid" value of about 100 μg per ml. The absorption curves of the products obtained when this reaction was carried out with several carbohydrates and with the protein-free filtrate from human milk were studied. They showed little support for the existence of glucuronic acid. Paper chromatography in a pyridine - *n*-pentanol - water (35:35:30) system showed no glucuronic acid in deproteinised milk, but it could not be recovered when added in quantities of less than 50 μg per ml before deproteinisation. Chromatograms run in butanol - acetic acid - water (70:7:23) showed a spot with the same R_F value as D-glucuronolactone, but this was shown to be due to L-fucose. When milk is deproteinised by ferric hydroxide with cooling during the process, a filtrate is obtained of pH between 6·5 and 7·3, which contains only traces of fucose. If trichloroacetic acid or tungstic acid is used as the deproteinising agent, large quantities of fucose are found. F. POWELL

947. **Specific methods for the micro-determination of pyridine nucleotides and pyridine nucleotide enzymes.** J. B. Jacobson and L. Astrachan (Johns Hopkins Univ., Baltimore, Md., U.S.A.). *Arch. Biochem. Biophys.*, 1957, **71** (1), 69–80.—Fluorimetric methods are described for the assay of about 10^{-8} mole of di- and of tri-phosphopyridine nucleotides in both their oxidised (fluorescent) and reduced (non-fluorescent) forms. The fluorescence of the oxidised form is developed with 5 *N* NaOH or with ethyl methyl ketone, and comparative measurements of fluorescence made before and after selective reduction with specific enzymes enables quant. estimation of the two nucleotides to be made. The reduced forms are similarly determined from fluorescence measurements after oxidation with three specific enzymes. Adaptations of the method for studying the course of specific enzyme reactions are described. W. H. C. SHAW

948. **Rapid method for the determination of lipid phosphorus.** C. E. Fischbach E. and C. A. López Pérez (Lab. Min. de Hacienda, Venezuela). *Acta Cient. Venezolana*, 1957, **8** (2), 40–42.—The method is based on destruction of organic matter

with H_2SO_4 - HNO_3 and pptn. of the P as ammonium molybdophosphate. The ppt. is dried at 120° to 130°. The time for a determination is 4 hr., with a mean relative error of 1·6% (8 results), compared with 13 to 15 hr. and a mean relative error of 1·0% (3 results) for the method of A.O.A.C. VII.

E. C. APLING

949. **Infra-red spectra of 3-phenyl-2-thiohydantoins of amino acids.** A. Epp (Prairie Reg. Lab., Saskatoon, Sask., Canada). *Anal. Chem.*, 1957, **29** (9), 1283–1287.—Infra-red spectra, characterising the 3-phenyl-2-thiohydantoins, the 3-(phenyl-*p*-azophenyl)-2-thiohydantoins and the 3-*o*-nitrophenyl-2-thiohydantoins of amino acids, are illustrated. The spectra of some preparations deviate from the characteristic phenylthiohydantoin absorption patterns. G. P. COOK

950. **Detection of tyrosine on paper chromatograms.** D. P. Schwartz and M. J. Pallansch (Eastern Res. and Devel. Div., Agric. Res. Service, U.S. Dept. of Agric., Washington, U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1561–1562.—Tyrosine is specifically detected on chromatograms of acid-hydrolysed proteins by exposure to the brown fumes generated by HNO_3 and Cu, followed by exposure to NH_3 fumes. The resulting yellow spot fades on exposure to air, but is renewed on re-exposure to NH_3. The test will detect 10 μg per sq. cm. After the gaseous reagents have disappeared from the paper, it may be sprayed with ninhydrin or other reagents. Solvents of the phenolic type interfere, and must be completely removed from the paper before testing. Several tyrosine deriv. gave strong reactions, and tryptophan a weak one, but all other naturally-occurring amino acids failed to react. R. E. ESSERY

951. **Thermo micro-methods for identification of glutamic acid antipodes.** A. Kofler, N. Delande and A. Lacourt (Microchem. Dept., Univ. Brussels, Belgium). *Microchem. J.*, 1957, **1** (1), 55–57.—A simple microscope technique for the identification of D(−)-, L(+)- and (±)-glutamic acid is proposed. The tests are based on the differences in crystal habits of the ppt. on evaporation of a dil. ethanolic soln. of the sample. If, on heating crystals of glutamic acid in a drop of liquid paraffin, water of crystallisation is observed, the sample is (±)-glutamic acid. This is confirmed if recrystallisation from dil. ethanol gives aggregates of needles. If these tests are negative, the sample is first mixed with L(+)-glutamic acid and recrystallised from dil. ethanol. If aggregates of needles characteristic of the hydrated (±)-isomers are formed, the original sample was D(−)-glutamic acid. If no needles are formed, the original sample is probably L(+)-glutamic acid. This can be confirmed by mixing it with the D(−)-isomer and recrystallising as described above; aggregates of (±)-compound needles should result. D. F. PHILLIPS

952. **Interferences with biuret methods for serum proteins. Use of Benedict's qualitative glucose reagent as a biuret reagent.** R. J. Henry, C. Sobel and S. Berkman (Bio-Sci. Lab., Los Angeles, Calif., U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1491–1495.—Results of the biuret method for the determination of serum proteins with Benedict's reagent agreed within experimental error with those of the Kjeldahl method. Of possible sources of interference, turbidity was best eliminated by extraction with ether; bilirubin did not interfere in the absence of turbidity. Haemolysis caused interference, but a correction can be applied if the haemoglobin is determined independently. Interference due to salts and ammonia varies with the particular biuret technique employed, but with Benedict's reagent little interference was noted with Na_2SO_3 (26%) and Na_2SO_4 (23%) if square cuvettes were used, round ones leading to slightly high results owing to refractive-index differences. If the ammonia N did not exceed five times the protein N, the error from it was insignificant. R. E. ESSERY

953. **Examination of the dioxopiperazine reaction of proteins.** Toshihiko Ohira and Kashio Imai (Dept. of Textile Chem., Fac. of Textiles, Shinshu Univ., Ueda, Nagano Prefecture). *J. Agric. Chem. Soc., Japan*, 1957, **31** (6), 413–416.—The detection of proteins by the dioxopiperazine reaction was examined with various proteins. On being treated with Na_2CO_3 soln. (1%) or $Ba(OH)_2$ soln. (3%), proteins containing S produce sulphide S. The intensity of the dioxopiperazine reaction of proteins increases with increasing concn. of S in proteins.

K. SAITO

954. **Toluene-*p*-sulphonic acid spot-plate test for steroids.** E. Epstein, W. O. Maddock and A. J. Boyle (Dept. of Med. and Chem., Wayne State Univ., Detroit, Mich., U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1548–1549.—The steroid (10 to 100 μg) is overlaid with 20 mg of crystalline toluene-*p*-sulphonic acid on a spot-plate, and heated to between 110° and 120°, being held there so that melting is complete within 3 to 4 min. Colour develops when melting begins, and deepens as heating proceeds. Colour and fluorescent reactions are tabulated for 29 steroids and compared with those given by conc. H_2SO_4. The reagent described does not char the steroid, and gives a greater variety of colour and more intense fluorescence than does H_2SO_4. As little as 3 μg of oestrone can be detected by colour, 0·5 μg by fluorescence.

R. E. ESSERY

955. **Method for the chemical estimation of oestradiol, oestrone and oestriol in the peripheral blood of women.** A. Puck (Univ.-Frauenklinik, Bonn, Germany). *Klin. Wochschr.*, 1957, **35** (16), 808–814.—Plasma (20 ml) is extracted with ether (3 × 15 ml); the ether extract is washed with H_2O (5 ml) and the solvent removed. The residue is dissolved in ethanol (10 ml), and H_2O (10 ml) is added slowly; the liquid is transferred to a separating funnel, rinsing with ethanol (10 ml) and then with H_2O (10 ml). The aq. ethanol is washed with light petroleum (2 × 40 ml), concentrated to 2 ml, mixed with benzene (38 ml) and extracted with *N* NaOH (3 × 30 ml). The combined NaOH soln. are acidified to pH 2 with 15 *N* H_2SO_4 and extracted with ether (3 × 30 ml). The ether is removed, the residue is dried thoroughly in a vacuum desiccator and then taken up in 95% ethanol. Quant. paper chromatography is then carried out as previously described (*Anal. Abstr.*, 1956, **3**, 800).

H. F. W. KIRKPATRICK

956. **Total neutral 17-ketosteroids. Clinical method for measurement.** M. S. Kafka and P. K. Bondy (Dept. of Internal Med., Yale Univ., New Haven, Conn., U.S.A.). *Clin. Chem.*, 1957, **3** (3), 178–184.—The method of Masuda and Thuline (*J. Clin. Endocrin. & Metabolism*, 1953, **13**, 581) is modified to simplify manipulation so that a larger number of determinations can be run at one time.

H. F. W. KIRKPATRICK

957. **Plate method of quantitative determination of pepsin activity.** L. Krówczyński and A. Smajkiewicz (Z. Zakładu Chem. Farm. A.M.w Lublinie). *Acta Polon. Pharm.*, 1957, **14** (1), 41–44.—A medium prepared from agar soln. (15 ml) and soln. of commercial casein in 0·2% NaOH (5 ml) is acidified with 10% HCl to pH 1·5 to 2·0. The suspension is poured into a 9-cm Petri dish and, when it hardens, five holes of 9-mm diam. are cut symmetrically in it. Into three holes is introduced 0·1 ml of 1%, 0·5% and 0·25% of pepsin of known strength in 0·5% HCl. Into the other two holes is introduced 0·1 ml of 1% (X) and 0·5% ($X/2$) of the pepsin under test. The plate is incubated at 40° for 12 hr. The clear regions of digested protein are then measured. The concn. of X and $X/2$ is read off from a graph of the standard pepsin concn. against diameter. The results of several hundred tests were found to be reproducible to within ±4%. B. LAKE

See also Abstracts 864, 881, 975, 1039.

Drugs

958. **The estimation of nornicotine formation in isolated tobacco root.** H. B. Schröter and L. Engelbrecht (Inst. Plant Culture, Sci. Acad., Berlin, Germany). *Arch. Pharm., Berlin*, 1957, **290** (4), 204–206.—Circular paper chromatography is used to separate nicotine, anabasine and nornicotine in tobacco-root extract. Washed roots, about 3 to 8 weeks old, are shredded and extracted with ethanol; the extract is acidified with dil. HCl and the ethanol is removed on a water bath. The acid soln. is then made alkaline with aq. NH_3 soln. and extracted with $CHCl_3$, and the soln. is applied to circular filter-paper. The alkaloids are separated by using a borate buffer soln. (pH 7·8) and a water-satd. *n*-butanol system. The separated constituents are identified with cyanogen bromide (according to Munier). G. R. WHALLEY

959. **Determination of theophylline and aminophylline by non-aqueous titration.** M. A. McEniry (Food and Drug Admin., Dept. of Health, Educ. and Welfare, St. Louis, Mo., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1957, **40** (3), 926–929.—The sample, containing about 150 mg of theophylline, is dissolved in dimethylformamide (25 ml) and titrated with 0·1 *N* Na methoxide in the presence of thymol blue. The method is more rapid and gives more accurate results than that of the U.S. Pharmacopoeia XV. A. A. ELDRIDGE

960. **Estimation of quercetin and rutin.** D. G. Constantinesco and R. Oteleanu (Inst. Badań. Farm. Kontrol., Leków, Bucharest). *Acta Polon. Pharm.*, 1957, **14** (1), 27–32.—The intensity of the colour produced on adding antimony trichloride to a methanolic solution of quercetin gives a measure of the quercetin present. Rutin is determined in the same way after hydrolysis to quercetin. B. LAKE

961. **Method for determination of mannitol hexanitrate.** M. A. McEniry (Food and Drug Admin., Dept. of Health, Educ. and Welfare, St. Louis, Mo., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1957, **40** (3), 929–932.—The sample is distilled with KOH in ethanol to remove ammonium salts and the residue, after reduction with Devarda's alloy and ethanol, is distilled with frequent addition of ethanol until 5 ml of distillate require not more than 1 drop of 0·01 *N* H_2SO_4 for neutralisation. The method is rapid and accurate; phenobarbitone does not interfere. A. A. ELDRIDGE

962. **Determination of the ascaridole content of chenopodium oil. New formula for the calculation of results using the method of Cocking and Hymas.** J. H. Helou. *An. Fac. Farm. Odont., Univ. S. Paulo*, 1955, **13**, 179–188.—The various factors suggested for the conversion of 0·1 *N* $Na_2S_2O_3$ (ml) to ascaridole in the method of Cocking and Hymas (*Analyst*, 1930, **55**, 180) for the assay of chenopodium oil are reviewed, and the factor 1 ml of 0·1 *N* $Na_2S_2O_3 \equiv 0{\cdot}005604$ g of ascaridole is proposed. This factor, when used with a correction formula, gives results in agreement with the quadratic expression of Beckett and Jolliffe (*Anal. Abstr.*, 1954, **1**, 361). The ascaridole content of the chenopodium oil must be at least 50% for application of this method. G. H. FOXLEY

963. **Solubility of antibiotics in twenty-four solvents: use in analysis.** P. J. Weiss, M. L. Andrew and W. W. Wright (Food & Drug Admin., Washington, D.C., U.S.A.). *Antibiot. & Chemother.*, 1957, **7** (7), 374–377.—As a guide to the selection of suitable solvents for the preliminary separation of antibiotics from mixtures and formulated products, solubilities of 32 different salts of 18 antibiotics in 24 solvents are listed. W. H. C. SHAW

964. **Identification and determination of NN'-dibenzylethylenediamine (benzathine) in benzathine penicillin.** R. Simões. *An. Fac. Farm. Odont., Univ. S. Paulo*, 1955, **13**, 85–89.—Two specific reactions for benzathine are given which can also be applied to the differentiation of benzathine penicillin and procaine penicillin. One is based on Liebermann's nitroso reaction and the other on the preparation of an iodo derivative. *Procedure* (*i*)—Dissolve the sample (0·015 g) in 0·2 *N* NaOH (2 ml) and extract with ether (5 ml). Evaporate the ether, dissolve the residue in 3 drops of HCl (10%), cool to 5°, add 3 drops of $NaNO_2$ soln. (10%) and set aside for 5 min. Dilute with H_2O (3 ml) and extract with ether (5 ml). Evaporate the ether and treat the residue with 2 drops of conc. H_2SO_4. A green colour is produced which, on addition of H_2O (0·2 ml), changes to pink and, on addition of 2 *N* NaOH (1 ml), changes to blue. *Procedure* (*ii*)—Dissolve the sample (0·001 g) in 0·01 *N* NaOH (2 ml) and extract with ether (3 ml). Evaporate the ether and treat the residue with a drop of benzaldehyde and a drop of iodine soln. Dark needles of 2-(benzylamino-1-(N-benzyl-N-iodoamino)di-iodoethane are formed, which are visible under the microscope. The benzathine content of benzathine penicillin may be determined by decomposition of the salt with NaOH, and extraction and recovery of the base, which can then be titrated with 0·1 *N* HCl or dissolved in excess of 0·1 *N* HCl and back-titrated with 0·1 *N* NaOH. Ten determinations with this latter method gave benzathine contents between 19·99 and 20·02%. G. H. FOXLEY

965. **Colorimetric determination of folic acid in presence of azo dyes.** S. K. Ganguly and Hrishikesh Bhattacharya (Bengal Immunity Res. Inst., Calcutta). *Indian J. Pharm.*, 1957, **19** (7), 170–173.—Folic acid may be determined in the presence of some azo dyes by the Bratton - Marshall method (*J. Biol. Chem.*, 1939, **128**, 537) provided that (*a*) the concn. of each does not exceed 35 μg in 2 ml, and (*b*) the dye is quant. reduced both by Zn plus HCl

and by ammoniacal H_2S. Since folic acid is not reduced by the H_2S under these conditions it may be estimated by the difference in colorimeter readings. The method is applicable to pharmaceutical preparations that are artificially coloured.
D. P. FELIX

966. **Determination of panthenol [pantothenyl alcohol] and pantothenates in multivitamin preparations.** M. Schmall and E. G. Wollish (Anal. Res. Lab., Hoffman-La Roche, Inc., Nutley, N.J., U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1509–1513.—By alkaline hydrolysis, panthenol and pantothenates yield β-alanol and β-alanine, respectively, which are then determined colorimetrically with Na 1:2-naphthaquinone-4-sulphonate or ninhydrin. Interfering substances, which include riboflavine, nicotinamide, amino acids, liver extracts and several metals, are removed by using combinations of ion-exchange resins and adsorbents, which vary according to the particular preparation being examined. The naphthaquinone and ninhydrin methods allow as little as 2·5 and 1 mg, respectively, to be determined in multivitamin prep. with a precision of $\simeq \pm 2\%$. The ninhydrin method is advocated, as occupying less time, requiring less critical pH adjustment in coloured soln., and permitting the assay of prep. of relatively low potency.
R. E. ESSERY

967. **Bioassay of insulin by the compensation method.** A. B. Macallum (Dept. of Biochem., Univ. of Western Ontario, London, Canada). *Canad. J. Biochem. Physiol.*, 1957, **35** (8), 599–604.—The basis of the method is to compensate for the lowering of the blood sugar by perfusing glucose - saline into the insulinised rabbit. The previously starved rabbits are placed in stocks and a blood sample is taken for blood-sugar estimation. Insulin is injected intravenously, followed by a slow continuous infusion of 4 or 6% glucose in normal saline from an adjustable motorised syringe. Further blood samples are taken at 20-min. intervals over 80 min., and these are pooled for blood-sugar estimation. The glucose utilisation is calculated from the observed blood-sugar levels by the use of a modified Bouckaert and de Duve equation (*Arch. Int. Pharmacodyn.*, 1944, **69**, 485). With four groups of nine rabbits it was found that the glucose utilisation was related to the insulin dosage. G. F. SOMERS

968. **Identification of barbiturates by paper chromatography.** P. Relyveld (Farm. Lab., Rijksuniv., Utrecht, Holland). *Pharm. Weekbl.*, 1957, **92** (18), 621–629.—A simple and rapid method comprises a chromatographic separation on Whatman No. 1 paper, with $CHCl_3$ - aq. NH_3 (2:1) as the mobile phase in a descending technique; this is followed by a second separation on paper buffered at pH 8, the stationary solvent being a citric acid - phosphate buffer, with $CHCl_3$ as the descending mobile phase. Spots can be clearly observed in u.v. light and can be marked by spraying the paper with NaOH soln. and then contact printing; as little as 10 to 25 μg can be identified in this way when known substances are used as markers.
P. HAAS

969. **Paper-chromatographic identification of barbituric acid derivatives in toxicological analysis.** J. Bäumler (Canton Lab., Basle, Switzerland). *Mitt. Lebensmitt. Hyg., Bern*, 1957, **48** (3), 135–145.—*Isolation*—Urine is acidified with dil. H_2SO_4 and extracted with ether, the ether extract is treated with charcoal if necessary and the ether removed. Other biological materials are extracted by the Stas - Otto process. The residue is dissolved in a little methanol and the soln. applied to Whatman No. 1 filter-paper. *Zwikker reaction*—After drying, the paper is sprayed lightly with a 2% soln. of a cobalt salt, dried at room temp. for 10 min. and placed in the chromatography chamber (see below) for 30 sec. A positive reaction is shown by a violet colour. *Paper chromatography*—This is carried out in an atmosphere equilibrated with 25% aq. NH_3, with *iso*propyl alcohol - $CHCl_3$ - 25% aq. NH_3 (6:3:1) as solvent. The time required under the prescribed conditions is 12 to 15 hr. *Development of the colour spot*—After drying at room temp. and then at 60° for 10 min. the paper is immersed in saturated $HgNO_3$ soln., when a bluish to brownish-grey colour forms after a minute. The paper is washed 3 times for 1 min. in H_2O and dried.
H. F. W. KIRKPATRICK

970. **X-ray powder diffraction patterns of some organic compounds.** D. H. Lennox (Occupational Health Div., Dept. of Nat. Health and Welfare, Ottawa, Ontario, Canada). *Anal. Chem.*, 1957, **29** (10), 1433–1435.—Data are presented for 3 sulpha compounds (sulphamerazine, sulphapyridine and sulphathiazole sodium), 2 chlorinated insecticides (aldrin and γ-chlordane), and the fungicide nabam and 4 related compounds. K. A. PROCTOR

971. **Microchemical identification of some antihistamine drugs.** E. G. C. Clarke (Physiol. Dept., Royal Vet. Coll., London). *J. Pharm. Pharmacol.*, 1957, **9** (11), 752–758.—Crystal and colour tests are described for microgram quantities of 50 antihistamine drugs. A. R. ROGERS

972. **The assay and identification of pyrimethamine and its preparations.** R. E. A. Drey (Wellcome Chemical Works, Dartford, Kent, England). *J. Pharm. Pharmacol.*, 1957, **9** (11), 739–746.—Pyrimethamine (**I**) and tablets of **I** are assayed gravimetrically by pptn. of the tungstophosphate from a boiling 0·04% soln. in 3% H_2SO_4. For the assay of tablets containing **I** and quinine dihydrochloride (**II**), prepare an extract by heating the powdered sample with 0·1 *N* HCl at 50° for > 1·5 hr., dilute suitably and measure the extinction at 270·5 mμ and at 347 mμ. The values of $E^{1\%}_{1\,cm}$ in 0·1 *N* HCl are—at 270·5 mμ, **I** 317, **II** 17·1; at 347 mμ, **I** 0·06, **II** 134·6. For the determination of **I** in powder containing **I** and sulphaguanidine (**III**), extract the sample by shaking with $CHCl_3$, filter and evaporate the extracts, dissolve the residue in 0·1 *N* HCl containing acetic acid, and measure the extinction at 272·5 mμ; $E^{1\%}_{1\,cm}$ at 272·5 mμ for **I** in 0·1 *N* HCl = 320. Determine the **III** either by the B.P. method or from the S content of the powder; **I** does not interfere in either procedure. Tests of identity for **I**, additional to those described in the B.P.C., are also given. A. R. ROGERS

973. **The quantitative measurement of *l*-phenylephrine hydrochloride in dilute aqueous solution.** E. L. Pratt (Winthrop Lab., Rensselaer, New York, U.S.A.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1957, **46** (8), 505–508.—In dil. aq. soln., phenylephrine is readily acetylated and the wt. of the diacetyl derivative and its specific rotation can be used to determine the proportion of the *l*-isomer of phenylephrine in the sample. The method affords a means of checking the stability of this compound during storage and its resistance to the effects of

heat, *e.g.*, when being sterilised. Final calculation of the result is based on a value of $-115{\cdot}4°$ for $[\alpha]_D^{25}$ of the derivative. D. G. FORBES

974. **A note on the assay of zinc stearate.** J. S. Faber (State Univ., Grote Rozenstraat 17, Groningen, Holland). *J. Amer. Pharm. Ass., Sci. Ed.*, 1957, **46** (8), 512–514.—The method of the U.S.P. for the assay of samples of Zn stearate gives low values for the content of zinc oxide when sodium or alkaline-earth stearates or free fatty acids are present as impurities. Three methods are proposed for the accurate determination of the Zn by complexometric titration with EDTA (disodium salt). D. G. FORBES

975. **Comparative *in vivo* methods for evaluating antacids in humans.** J. W. E. Harrisson, D. D. Abbott, J. I. Feinman and E. W. Packman (LaWall and Harrisson Res. Lab., Philadelphia, Pa., U.S.A.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1957, **46** (9), 549–552.—A continuous recording device constructed from commercial components is described for the accurate determination of gastric pH without removal of gastric juice from the stomach. A polyethylene shield prevents contact between the glass electrode and gastric mucosa. A. R. ROGERS

976. **A method for the measurement of lubricating property of medicinal products.** G. Levy and T. W. Schwarz (School of Pharm., Calif. Univ., San Francisco, U.S.A.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1957, **46** (9), 558–561.—An instrument ("lubrimeter") is described which measures the lubricant effect of medicinal products under conditions approaching normal use. A polished brass roller is rotated at a low constant speed. On it rests a Bakelite drum which can be covered with the test surface; the drum is free to rotate around its own axis. The roller is partly immersed in a reservoir of lubricant which is fitted with a scraper, so that as the roller rotates it picks up a film of uniform thickness on the surface. Friction causes rotation of the drum which is opposed by a calibrated spring and after a few minutes an equilibrium position is reached. A low friction value indicates a good lubricant. A. R. ROGERS

See also Abstracts 770, 793, 857, 1013.

Food

977. **Microscopic characteristics of starches in the identification of ground cereal grains.** M. M. MacMasters, M. J. Wolf and H. L. Seckinger (Northern Utilization Res. and Development Div., Agric. Res. Service, Dept. of Agric., Peoria, Ill., U.S.A.). *J. Agric. Food Chem.*, 1957, **5** (6), 455–458.—The microscopic appearance of ungelatinised and gelatinised starch granules of common commercial grains (maize, wheat, grain sorghum, barley, oats, rye and rice), and of endosperm and pericarp particles, is discussed as a useful means of identification. Various preparations are illustrated. S.C.I. ABSTR.

978. **Use of the Carrez clearing reagent in polarimetric determination of starch.** I. Sarudi (v. Stetina) (Chem. Untersuchungsamt, Szeged, Hungary). *Z. LebensmittUntersuch.*, 1957, **106** (2), 142–144.—In the determination of starch in foods by the Ewers - Grossfeld and the Grossfeld and Baumann methods, molybdophosphoric acid can be replaced, without loss of accuracy, by the cheaper and more convenient Carrez reagent. In the determination of starch in cinnamon, satisfactory clearing can be obtained with the Carrez reagent, but not with the molybdophosphoric acid reagent. P. S. ARUP

979. **Measurement of enzymatic amylolytic activity. Group A. IV. Methods depending on the determination of the iodine - starch reaction (examination of the starch breakdown by the [colour] change of the iodine reaction).** H. Wildner and G. Wildner (Ireks Forschungsinst. für Gärungswissenschaft, Kulmbach, Germany). *Brauwissenschaft*, 1957, **10** (4), 90–101.—Methods of determining amylases by iodine colorimetry are reviewed and discussed.

Group A. V. Methods based on the determination of the decrease in volume or weight of starch. (Determination of amylases by alcoholic precipitation of the substrate). H. Wildner and G. Wildner. *Ibid.*, 1957, **10** (6), 160–162.—Appropriate procedures are discussed for determining (*a*) the difference in quantity of alcohol-precipitable starch before and after enzyme action; (*b*) the decrease in vol. of sediment of an alkaline meal suspension; (*c*) the loss in wt. of initial starch quantity and insol. residue after enzyme activity ceases.

Group B. I. Chemical methods. H. Wildner and G. Wildner. *Ibid.*, 1957, **10** (6), 162–168; (7), 189–194.—The literature on the determination of reducing substances resulting from the hydrolysis of starch is reviewed, with special reference to the methods of Kjeldahl and Bertrand, Willstätter and Schudel, Hagedorn and Jensen, and Blom and Rosted; and Lintner's work and modifications by other authors on the fermenting ability of malt are surveyed. S.C.I. ABSTR.

980. **Chemical method for the determination of histamine in canned tuna fish.** O. S. Sager and W. Horwitz (Div. Food, Food and Drug Admin., Dept. of Health, Educ. and Welfare, Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1957, **40** (3), 892–904.—Histamine (which results from decomposition) is extracted from the sample in a high-speed blender; an aliquot of the filtered extract is treated with benzaldehyde and NaOH, and the resulting benzylidenehistamine is extracted with benzene - *n*-butanol (3:2, v/v). The soln. is passed through a column of cotton washed and dried after being soaked in a soln. of Na acetate (5 g) and succinic anhydride (40 g) in acetic acid (300 ml). The histamine is eluted with 0·4 *N* H_2SO_4 followed by H_2O, and converted into a dye with diazotised *o*-nitroaniline. The dye is extracted with *iso*butyl methyl ketone and treated with barbitone sodium soln. (10 g in 1 litre of water), adjusted to pH 7·7 with acetic acid, to convert interfering red colours to yellow. The histamine is then determined spectrophotometrically at 475 mμ. If the acid extracts in the bio-assay (with a segment of guinea-pig ileum) are neutralised with $NaHCO_3$ and Ringer - Locke soln. instead of with Na_2CO_3, the results of the biological and chemical methods are in agreement. Fish in organoleptic classes 3 and 4 (*cf.* Hillig, *Ibid.*, 1956, **39**, 773) may contain 0 to 736 mg of histamine per 100 g. A. A. ELDRIDGE

981. **Rapid complexometric method for the determination of the calcium content of milk.** C. W. Raadsveld (Netherlands Inst. for Dairy Research, Ede, Holland). *Neth. Milk Dairy J.*, 1956, **10**, 114–122.—The determination of Ca in milk (10 ml diluted to 500 ml) by direct titration with 0·1 *M* EDTA (disodium salt), with murexide as indicator, can be carried out similarly to that of Ca in water or blood serum. The difficulty due to turbidity is overcome by titrating to the same tint as that of a soln. prepared by adding to a similarly diluted sample a mixed indicator of phenolphthalein and bromocresol purple (7 ml) and 2 drops of 4 *N* NaOH. The milk phosphates do not interfere with the determination. The results for Ca show satisfactory reproducibility and are $\geq 2\%$ higher than those obtained gravimetrically. The use of an excess of EDTA followed by back-titration with standard $CaCl_2$ soln. offers no advantages.

P. S. ARUP

982. **Detection and determination of cows' milk in fresh sheep's-milk cheese.** Z. von Sándor and A. Péter (Hungarian State Control Station for Milk Products, Budapest, Hungary). *Z. Lebensmitt-Untersuch.*, 1957, **106** (2), 106–109.—The determination is made on the fresh raw cheese (used as a base for Liptauer cheese) which has first been salted with 2% of NaCl and kept for one day, or on the salted Trappist sheep's-milk cheese. A prep. is made of the water-insol. defatted casein under standard conditions, and the solubility of this prep. (0·3 g) in 99·7% acetic acid (20 ml) after heating for 20 min. on a steam bath and then cooling is determined. Sheep's-milk casein is completely sol., but cows'-milk casein is 50% insol. The insol. matter is washed with ethanol, collected, dried, and weighed. A graph is given from which the percentage of cows' milk can be found from the insol. matter (mg) obtained. P. S. ARUP

983. **Determination of egg content of egg-dough products.** L. Acker and W. Diemair (Univ. Inst. für Lebensmittelchemie, Frankfurt am Main, Germany). *Z. LebensmittUntersuch.*, 1957, **105** (6), 437–445.—Various methods for determining the egg content of egg-dough products, *e.g.*, macaroni, dough pastes of vermicelli and dumpling types, were critically examined. The method of Hadorn and Jungkunz was most suitable; that of Riffart and Keller yielded occasional low values without detectable cause. Data are given on the lipid and sterol contents, total lipids (obtained by ethanol-benzene extraction and purified with ether), and comparison of results of different methods. Rapid titration methods were suitable for rough estimates only. S.C.I. ABSTR.

984. **Determination of caffeine in "caffeine-free" coffee.** H. Hadorn and H. Suter (Lab. VSK, Basel, Switzerland). *Mitt. Lebensmitt. Hyg., Bern*, 1957, **48** (2), 63–79.—Cortes's method (*Rev. Soc. Brasil. Chim.*, 1933, **4**, 105; *Z. LebensmittUntersuch.*, 1937, **74**, 106) of setting free the caffeine with conc. H_2SO_4, followed by extraction with $CHCl_3$ and decolorising with charcoal, has been improved by purifying the crude caffeine by chromatography, and the product is determined by a modification of Richter's colorimetric method based on the isolation of the caffeine as the periodide (*Z. Lebensmitt-Untersuch.*, 1954, **98**, 107). This enables the caffeine in 2 to 3 g of caffeine-free coffee to be accurately determined. P. HAAS

985. **Determination of shell constituents in cacao products.** H. van Brederode and C. J. Reeskamp (Chem. Lab., N. V. Cacaofabriek "De Zaan", Koog-Zaandijk, Holland). *Z. LebensmittUntersuch.*, 1957, **105** (6), 461–473.—Methods were compared, in particular the determination of pectin, of crude fibre, and the microscopical count of sclerenchymatous cells, and the results were examined statistically. The last method is preferred, but the degree of grinding of the meal must be taken into consideration. S.C.I. ABSTR.

986. **Determination of nitrogen in barley and malt.** J. R. Heron (Messrs. Heron & Comrie, 45, City Rd., London). *J. Inst. Brewing*, 1957, **63** (4), 339–340.—The observed differences in N content of the same barley or malt, both within and between laboratories, are ascribed to fractionation during the weighing out of the ground sample. It is shown that when barley is ground to a normal texture and the grist separated with a 20-mesh sieve, the coarse fraction contains more, and the fine fraction less, N than the mean given by a finely ground sample of the same barley, this being ascribed to retention of more aleurone layer by the coarse particles. With malt, the reverse is the case, the coarse particles, which are almost pure husk, containing less N than the fine fraction. If care is taken to avoid fractionation during weighing out, results can be as accurate with a normal grind as with a fine one, but to minimise risk of such fractionation, grinding as finely as possible is recommended, with a double grind for barley.

R. E. ESSERY

987. **Comparative studies of methods of hop analysis. III. Recent methods of analysis for α-acids and other resin constituents.** L. R. Bishop (Stag Brewery, Pimlico, London). *J. Inst. Brewing*, 1957, **63** (4), 316–330.—The results are presented of a collaborative study of four methods, involving some 300 analyses in 16 laboratories. The methods studied were (*i*), the silica-gel polarimetric method of Verzele and Govaert (*Wallerstein Labs Commun.*, 1954, **17**, 119); (*ii*), the charcoal polarimetric method of Gough (*cf. Anal. Abstr.*, 1956, **3**, 2297); (*iii*), the spectrophotometric method of Alderton *et al.* as modified by Klopper (*cf. Anal. Abstr.*, 1954, **1**, 3138; 1955, **2**, 3208); and (*iv*), the conductimetric method of Hartong *et al.* (*Int. Tijdschr. Brouw. en Mout.*, 1956–1957, 33), involving conductimetric titration of a methanolic extract with methanolic Pb acetate. Precautions necessary during sampling and disintegration are described. A macerator is preferable to a mincer for disintegration, but the period of maceration is probably critical, and should be established for each mill. For use with the Ford and Tait and similar methods, a method of weighing the hops into a polyethylene bag, in which they are broken up, decanted into the extractor, and the bag cut up and added, is recommended. Toluene can replace benzene as a solvent, with reduction in hazard to health. All four methods studied are at least as accurate as established ones, and in none were systematic errors detected. The polarimetric methods seem to be rather more accurate than older ones, and the spectrophotometric and conductimetric methods may prove to be so with improvement in details. The polarimetric and spectrophotometric methods are reliable for old hops. All the methods, in particular the spectrophotometric and conductimetric, are quicker than the older ones.

R. E. ESSERY

988. **Method for the direct ethylenediaminetetra-acetate titrimetric determination of magnesium and calcium in beer.** J. L. Owades, G. Rubin and M. W. Brenner (Schwarz Lab. Inc., 230 Washington St., Mt. Vernon, N.Y., U.S.A.). *Proc. Amer. Soc. Brew. Chem., Ann. Mtg,* 1956, 193–201.—The method described is claimed to be rapid, precise and accurate. *Procedure*—The Ca plus Mg is determined by adding water (40 ml) and Na_2S soln. (2% w/v) (1 ml) to a de-gassed sample (5 ml), followed, immediately before titration with EDTA (disodium salt) soln. (0·1%) to the greenish-grey colour of the indicator, by buffer soln. (67·5 g of NH_4Cl in 400 ml of water, adjusted to pH 10 with aq. NH_3 and diluted to 1 litre) (5 ml) and Eriochrome black T indicator (0·2 g of dye in 50 ml of methanol containing 2 g of hydroxylamine hydrochloride) (10 drops). Magnesium is determined by pptg. the Ca in a 25-ml sample of de-gassed beer with a saturated soln. of ammonium oxalate (0·5 ml), keeping at 0° for 2 hr., filtering (without washing the ppt.) and titrating a 5-ml aliquot of the filtrate as before. Solid brewing materials, *e.g.*, hops, malt (1 to 2 g), are first wet-ashed with HNO_3, H_2SO_4 and H_2O_2, the clear solution is adjusted to pH 5 with the buffer soln., and diluted to 100 ml with water. Aliquots are titrated as before. S. C. JOLLY

989. **Oxidimetric determination of sorbitol in wine with the use of paper-chromatographic methods.** K. Täufel and K. Müller (Inst. für Ernährungsforsch., Potsdam-Rehbrücke, Germany). *Z. Lebensmittl Untersuch.*, 1957, **106** (2), 123–128.—The limitations of direct titration with potassium periodate, and of the chromatographic separation of sorbitol and glucose, are pointed out. *Procedure*—Fermentable sugars are first removed by incubation with a yeast suspension and, after removal of ethanol and insol. matter, a rough estimation of the sorbitol present is made by paper chromatography with the use of *n*-butanol - acetone - H_2O as ascending solvent, the sorbitol spot being revealed by spraying according to the method of Wolfrom and Miller (*Anal. Abstr.*, 1956, **3**, 3088). A chromatogram is then started with an appropriate vol. of the fermented soln. (distributed in parallel runs) according to the procedure described by Täufel *et al.* (*Brit. Abstr. C*, 1952, 316), the sorbitol being determined in the isolated spots by titration with potassium periodate. The method is accurate to within $\pm$ 3%, and allows 20 mg of sorbitol in 1 litre of wine to be detected. P. S. ARUP

990. **Determination of added distinctive cations in whisky. IX. Ultra-violet spectrophotometric determination of cerium.** M. J. Pro and R. A. Nelson (Alcohol and Tobacco Tax Div. Lab., Internal Revenue Service, Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1957, **40** (3), 932–936.—The whisky (50 ml) is evaporated to dryness and the organic matter is burned off. The residue is heated to fuming with H_2SO_4 (1 ml); then water (5 ml) and K_2CO_3 soln. (500 g in 500 ml of water; 5 ml) are added, and the extinction of the sample is measured against that of a reference (K_2CO_3) soln. at 340 mμ. The standard soln. of Ce ($\simeq$ 1000 p.p.m.) is prepared by dissolving 2·6590 g of $CeCl_3.7H_2O$ in water (1 litre). There is no interference from Fe up to 0·5 p.p.m. For 0·1 to 1·0 p.p.m. of Ce, the results are accurate to within 0·1 p.p.m. A. A. ELDRIDGE

991. **Paper-chromatographic investigation of vanillin and ethylvanillin in foodstuffs.** J. Deshusses and F. Desbaumes (Lab. Cantonal de Chimie, Geneva, Switzerland). *Mitt. Lebensmitt. Hyg., Bern*, 1957, **48** (2), 49–58.—Modifications of earlier chromatographic methods for detecting vanillin and allied compounds were employed for the examination of foodstuffs sold in Switzerland. The R_F values of vanillin, ethylvanillin and *p*-hydroxybenzaldehyde were re-determined and results considerably lower than those recorded by earlier workers were obtained. Liquid foodstuffs are first freed from ethanol and then defecated with basic Pb acetate and extracted with ether; the vanillin and allied aromatic compounds are separated from this extract with 2% aq. NH_3, the soln. is acidified, and the aldehydes are extracted with ether. After evaporation of the ether, the residue is dissolved in ethanol for chromatographic analysis. Solid foodstuffs are first extracted with 50% ethanol and, after removal of this solvent, defecation and further purification are carried out as described above for liquids. Ascending chromatography is carried out with Whatman No. 1 paper and *iso*butyl alcohol saturated with 2% aq. NH_3 as solvent. After being left overnight, the paper is air-dried and then sprayed with a 1% soln. of hydrazine sulphate in *N* HCl. After drying, the aldehydes are revealed as yellow spots on a white background which fluoresce in u.v. light. The *p*-hydroxybenzaldehyde has a characteristic fluorescence distinguishing it from vanillin and ethylvanillin. P. HAAS

992. **Microscopical detection of spices and other plant elements in foods.** E. Hanssen, R. Ihlenfeldt and G. Florian (Lab. der H. Bahlsens Keksfabrik K.G., Hannover, Germany). *Z. Lebensmittl-Untersuch.*, 1957, **105** (5), 361–373.—The value of microscopical-histological analysis for the detection of plant elements in foods is discussed. Descriptions are given of the necessary preliminary prep., *e.g.*, of sausages, baked products, jams, spice or herbs. Photomicrographs (29) are given showing the very distinctive structure of stone cells, epidermal tissue, stomata, hairs, etc., of currants; sclerenchyma, etc., of apricots; cuticular fissures, wood fibres, etc., of paprika; and hypodermal cells and wood parenchyma of vanilla-fruit wall, etc. S.C.I. ABSTR.

993. **Detection of the presence of small amounts of turmeric in other spices.** S. N. Mitra and S. C. Roy (West Bengal Public Health Lab., Calcutta). *J. Instn Chem., India*, 1957, **29** (3), 155–157.—The spice is extracted with ether and the usual boric acid test is applied to the extract. The colouring matter of turmeric is separated on a paper chromatogram by a suitable solvent, *e.g.*, *iso*butyl alcohol - ethanol - H_2O (4:1:2). O. M. WHITTON

994. **Identification and determination of boric acid and benzoic acid in egg products.** M. E. Stas (Rijks Inst. v. Volksgezondheid, Utrecht, Holland). *Chem. Weekbl.*, 1957, **53** (40), 536–538.—The official method for the identification of benzoic acid in meat is suitable for use with egg products. It is an improvement to centrifuge the ethanolic extract instead of filtering. For the determination of boric acid, the official method for liver sausage is suitable, but the amount of NaOH used must be increased. For the determination of benzoic acid, a mixture of the egg product with water is cleared with $CuSO_4$ and NaOH. An aliquot of the filtrate is concentrated and extracted with benzene after acidifying

with phosphoric acid. The benzene soln. is washed with water and titrated with *N* NaOH.

G. BURGER

995. **Separation and identification of four antioxidants, butylated hydroxyanisole, butylated hydroxytoluene, *n*-propyl gallate and nordihydroguaiaretic acid, by paper chromatography.** L. C. Mitchell (Div. Food, Food and Drug Admin., Dept. of Health, Educ. and Welfare, Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1957, **40** (3), 909–915.—The four substances used as antioxidants in food products can be separated by three alternative chromatographic procedures. In the first procedure, the paper is pre-impregnated by spraying with a 10% soln. of soya-bean oil in diethyl ether. Methanol containing 20% of water is employed as the first mobile phase (perpendicularly) and water as the second mobile phase (horizontally). The chromatogram is then sprayed with a 0·5% soln. of molybdophosphoric acid in ethanol and is immediately exposed to gaseous NH_3. In the second procedure, the chromatogram is developed first with aq. methanol (20% H_2O) to two-thirds of the normal distance and then, after air-drying, fully with water. In the third procedure, methanol containing 60% of water is used for development to the normal distance. A. A. ELDRIDGE

996. **Note on inhibition of growth response by heavy inocula in the assay of vitamin B_{12} with *Lactobacillus leichmannii*.** H. Lichtenstein and H. Reynolds (Human Nutrition Res. Div., Agric. Res. Service, Beltsville, Md., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1957, **40** (3), 993–995.—In the U.S.P. (XIV, 3rd Suppl., 1952) - A.O.A.C. (Krieger, *J. Ass. Off. Agric. Chem.*, 1955, **38**, 711) method for the assay of vitamin B_{12} with *Lb. leichmannii*, dilution of the inoculum to a concn. of less than 0·5 to 0·75 mg of dry cells per 10 ml may be necessary for optimal growth when the inoculum is dispensed one drop per assay tube. A. A. ELDRIDGE

997. **Analysis of fat-soluble vitamins. III. Determination and adjustment of the activity of adsorbents for chromatography and of the eluting power of elution solvents by means of the "shake test".** K. J. Keuning, G. J. van Dijk and M. J. Wiggers de Vries (N.V. Philips Roxane, Weesp, Netherlands). *Rec. Trav. Chim. Pays-Bas*, 1957, **76** (8), 747–756 (in English).—For chromatographic purposes, the activity of an adsorbent can be determined by shaking a weighed amount (2 g) of the adsorbent with a measured vol. (10 ml) of the elution solvent in which the substance to be eluted (*e.g.*, 1000 i.u. of vitamin A) has been dissolved; the activity is suitable if about 50% is adsorbed. The method is considered to be more accurate and convenient than the use of calorimetry or gas or dye adsorption. It is not necessary for the shake test that the compound to be determined be available in a pure form; thus the unsaponifiable matter from a fish-liver oil gave the same result as pure vitamin-A alcohol. The technique can also be used for finding the best composition of the elution solvent. A. R. ROGERS

998. **Analysis of fat-soluble vitamins. II. A chemical routine determination of vitamin D. Correlation with the biological determination.** F. J. Mulder, J. R. Roborgh, T. J. de Man, K. J. Keuning and K. H. Hanewald (N.V. Philips Roxane, Weesp, Netherlands). *Rec. Trav. Chim. Pays-Bas*, 1957, **76** (8), 733–746 (in English).—A colorimetric method is described for the determination of vitamin D_2 (ergocalciferol) and D_3 (cholecalciferol) in crystalline material, irradiation products, emulsions, solutions and dry products. The method, which is based on published procedures, allows for interference by fatty oils, unsaturated acids and other steroids, but vitamins A must be absent. The results agree with those by rat or chick assays. The coefficient of variation is $\simeq 2\%$. A single determination requires $\simeq$ 200,000 i.u. of vitamin D. The steps in the procedure are (*a*) saponification and extraction of the unsaponifiable material, (*b*) removal of tachysterol by maleic anhydride addition, (*c*) removal of interfering steroids by digitonin pptn., (*d*) colorimetry with $SbCl_3$ reagent, (*e*) correction for residual colour due to polyenes.

A. R. ROGERS

999. **Colorimetric estimation of vitamin D.** P. N. Luthra and J. N. Tayal [Ministry of Defence (CGDP), Kanpur, India]. *J. Pharm. Pharmacol.*, 1957, **9** (11), 784.—For the determination of vitamin D in tablets, oily soln. or injections with Nield - Russell - Zimmerli reagent ($SbCl_3$ and acetyl chloride in $CHCl_3$), the concn. of acetyl chloride in the reagent should be within the range 2·5 to 3·0%, and extinction readings should be taken at 30-sec. intervals during 6 min. A. R. ROGERS

1000. **Identification of stored-products insects by the micromorphology of the exoskeleton. IV. Adult legs.** J. J. Thrasher and O. L. Kurtz (Food and Drug Admin., Dept. of Health, Educ. and Welfare, Cincinnati, Ohio, U.S.A.). *J. Ass. Off. Agric. Chem.*, 1957, **40** (3), 973–991.—The microscopic appearance of leg segments of various grain and fruit beetles is described and illustrated.

A. A. ELDRIDGE

See also Abstracts 836, 872, 943, 946, 1014.

Sanitation

1001. **Ultra-micro determination of sulphides in air.** M. B. Jacobs, M. M. Braverman and S. Hochheiser (Dept. of Air Pollution Control, New York, N.Y., U.S.A.). *Anal. Chem.*, 1957, **29** (9), 1349–1351.—The method described is suitable for the determination of sulphides in concn. as low as a few parts per U.S. billion. Air is bubbled through an alkaline suspension of cadmium hydroxide in a Greenburg - Smith impinger. Under these conditions the pptd. sulphide is not oxidised by the stream of air. The concn. of CdS produced is determined by the methylene blue method.

G. S. ROBERTS

1002. **Micro-determination of dissolved oxygen in water. V. Development of a self-testing and fully compensating analytical method.** E. C. Potter and J. F. White (Central Elect. Authority, Boiler Corrosion Lab., St John's Wood, London). *J. Appl. Chem.*, 1957, **7** (8), 459–467.—Traces of Fe^{2+} are shown to interfere in the determination of micro quantities of dissolved oxygen in water samples. It is possible to eliminate the interference by passing the sample through a column of Zeo-Karb 225, provided that care is taken to establish equilibrium between the resin and sample with respect to oxygen. The procedure for the micro-determination of dissolved oxygen, with the techniques developed in previous papers (*Anal. Abstr.*, 1958, **5**, 277), is described. D. G. FORBES

1003. **Colorimetric determination of nitrate in water as Orange I.** K. R. Middleton (Reg. Agric. Res. Sta., Samaru, Zaria, Nigeria). *Chem. & Ind.*, 1957, (**34**), 1147–1148.—Details of a procedure are given in which the Griess reaction for NO_3^- determination is adapted for the absorptiometric estimation of NO_3^- in water. The method developed for reduction of the NO_3^- gives 100% recovery, but the reduction efficiency and also the colour intensity decrease slightly with rise in temp. *Procedure*—Measure an aliquot of the sample containing about 0·2 p.p.m. of nitrate N into a conical flask and add aq. NH_3 (sp. gr. 0·91) (2 ml), $MnSO_4$ soln. (1% in 5% acetic acid) (1 ml) and pure zinc dust (0·2 g). Stopper the flask and shake it vigorously for 5 min. Filter the mixture and place some of the filtrate (20 ml) in a boiling-tube, add the reagent (0·1% of sulphanilic acid and 0·08% of 1-naphthol in 75% acetic acid) (5 ml), mix and set aside for 30 min. Measure the extinction of the soln. in 2-cm cells, with a No. 602 filter. The results are referred to a calibration curve prepared by treating, in a manner similar to the sample, 0-, 1-, 2- and 3-ml portions of a standard soln. of KNO_3 (0·00361%) in 0·5 *N* aq. NH_3 soln. after initial dilution to 50 ml.

D. G. Forbes

1004. **Determination of polycyclic aromatic hydrocarbons in oysters collected in polluted water.** H. J. Cahnmann and M. Karatsune (Nat. Inst. Health, Bethesda, Md., U.S.A.). *Anal. Chem.*, 1957, **29** (9), 1312–1317.—A methanol extract of the oysters was fractionated by solvent partition with *cyclo*hexane, alkaline hydrolysis and a series of chromatographic separations on the non-saponifiable matter. The fractions were investigated spectrophotometrically and the eight hydrocarbons found, with their approximate concn., are listed. The total content was about 1 mg per kg of shucked oysters.

G. P. Cook

1005. **Quantitative determination of volatile acids by paper chromatography for application to sewage-sludge digestion.** R. M. Manganelli and F. R. Brofazi (Dept. of Sanitation, Rutgers Univ., New Brunswick, N.J., U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1441–1443.—The C_2 to C_4 fatty acids are completely separated as their ethylamine salts by descending chromatography at 20°, with a specified *n*-butanol - water - ethylamine solvent system. On dipping the chromatogram into chlorophenol red, blue spots, stable for 3 hr., appear on a yellow background. The area of the spot is linearly related to the concn. of acid, and regression equations are given for the calculation of the individual acids. For concn. ranging from 3 to 8 μg per μl, average errors ranged from ± 3 to ± 6%. (See also Roberts, *Anal. Abstr.*, 1958, **5**, 867.)

R. E. Essery

1006. **Colorimetric determination of tin in industrial wastes and receiving waters.** W. W. Sanderson and A. M. Hanson (Div. of Lab. and Res., New York State Dept. of Health, Albany, N.Y.). *Sewage Ind. Wastes*, 1957, **29** (4), 422–427.—The dithiol method, with Na lauryl sulphate as dispersant for stabilising the dithiol - Sn complex, has been adapted for use with the Beckman DU spectrophotometer. The use of 5-cm cells enables concn. of 0·01 to 0·18 mg to be determined, and, for a 100-ml sample, a sensitivity of 0·01 mg per litre can be attained. A simplified distillation procedure for isolating Sn from industrial wastes is described.

S.C.I. Abstr.

1007. **Determination of phenolic-type compounds in water and industrial waste waters. Comparison of analytical methods.** E. F. Mohler, jun., and L. N. Jacob (Sun Oil Co., Toledo, Ohio, U.S.A.). *Anal. Chem.*, 1957, **29** (9), 1369–1374.—The five most widely used methods for determining trace amounts of phenolic-type compounds in water and waste waters were evaluated. The aminophenazone procedure was the fastest, most precise and accurate colorimetric method, even for the 0 to 100 parts per thousand million range. The u.v. absorption method offers considerable promise for determining all phenolic-type compounds found in refinery waste waters in both the parts per million and parts per thousand million ranges. Comprehensive data for all the methods are tabulated.

G. P. Cook

1008. **Titrimetric determination of warfarin in commercial preparations.** K. Kámen (High School Chem. Technol., Prague, Czechoslovakia). *Chem. Průmysl*, 1957, **7** (9), 472–476.—*Procedure*—Dissolve the sample (2·5 to 4 g) in 2 *N* NaOH (50 ml), filter, and dilute with 2 *N* NaOH to 50 ml. Add iodine (2 g) and filter. Dissolve the ppt. in ethanol (40 to 50 ml), add 0·1 *N* $AgNO_3$ (40 ml) and conc. HNO_3 (10 ml). After 30 min. titrate with 0·1 *N* NH_4SCN.

J. Böswart

See also Abstract 1020.

Agriculture and Plant Biochemistry

1009. **Mobilities of some polyols, sugars, acids and other compounds. Reactions on paper with the Godin reagent.** M. G. Lambou (U.S. Dept. of Agric., New Orleans, La., U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1449–1451.—The solvent system *n*-propanol - ethyl acetate - water (7:1:2) has been used to separate carbohydrates qual. and quant. from de-ionised extracts of sweet potato. Mobilities after development for 21 hr. at 25·5° are tabulated for 57 compounds, together with the colours given by the Godin reagent (*cf. Anal. Abstr.*, 1954, **1**, 2701) in daylight and u.v. light, and the fluorescence properties of the spots. The Godin reagent compared favourably in sensitivity with ammoniacal $AgNO_3$, and offered advantages in colour differentiation and the detection of minute quantities under u.v. light.

R. E. Essery

1010. **Determination of phytol and phytadiene and properties of pure standards.** E. Knobloch, F. Jančík, V. Janata, E. Kraus, D. Němcová and Z. Bacík (Res. Inst. Pharm. and Biochem., Prague, Czechoslovakia). *Chem. Listy*, 1957, **51** (7), 1379–1381.—Various methods for the determination of phytol (**I**) and phytadiene (**II**) were studied and quant. acetylation in pyridine medium and evaluation by means of infra-red measurements was found to be suitable. *Acetylation*—Weigh **I** (0·3 g) into a small vessel (20 ml), add acetylation reagent (12 g of acetic anhydride and 88 g of dry pyridine) (1·5 g), close, warm on the water bath (10 min.), then cool, and dilute with H_2O. Titrate with 0·5 *N* NaOH to phenolphthalein. Make a blank determination. For infra-red measurements the following solvents were used—for **I**, chloroform (990 cm^{-1}) or CCl_4 (3360 cm^{-1}); for **II**, *cyclo*hexanol (890 cm^{-1}). The preparation of pure standards of **I** and **II** is described.

J. Zýka

1011. **Separation and identification of aromatic acids in plant tissues by paper chromatography.** L. A. Griffiths (Imperial Coll. of Tropical Agric., Trinidad, B.W.I.). *Nature*, 1957, **180**, 286–287.—The solvent system benzene - acetic acid - water (6:7:3) enables the substituted cinnamic acids found in plants to be separated by paper chromatography. The system is extremely sensitive to temp. changes and after careful preparation (described) the chromatography is carried out for 3 to 4 hr. by ascending technique on Whatman No. 1 paper in a room maintained at 15°. The spots obtained are sufficiently well defined for a second chromatogram to be run at right angles, and may be detected and identified by their u.v. fluorescence and their colour with diazotised *p*-nitroaniline.
E. J. H. BIRCH

1012. **Determination of boron in fertilisers by ion exchange and flame spectrophotometry.** E. Bovay and A. Cossy (Stations Fédérales d'Essais Agricoles, Lausanne, Switzerland). *Mitt. Lebensmitt. Hyg., Bern*, 1957, **48** (2), 59–63.—*Procedure*—The sample (20 g) is shaken for 30 min. in a 1-litre flask containing 950 ml of water. The pH is adjusted to 4 and, after a further 30 min. shaking, the contents of the flask are made up to the mark, and filtered through a dry filter. A 50-ml aliquot (≡ 1 g of fertiliser) is passed through cation- and anion-exchange resins (H^+ and $HCOO^-$ forms, respectively) at approx. 3 to 4 ml per min., the column is eluted with water, and the eluate is made up to 200 ml. The eluate is examined in a Beckman DU spectrophotometer, with an oxy-hydrogen flame, at 548 mμ. Formic acid is added to the standard soln. to compensate for that taken up by the sample in passing through the anion-exchange resin. It was shown by a series of trials that, for fertilisers containing ≃0·5% of B, the effect of formic acid on the flame-photometric determination of B is negligible.
P. HAAS

1013. **Colorimetric determination of chlortetracycline hydrochloride in feed supplements, veterinary therapeutics and food preservatives.** F. S. Chiccarelli, M. H. Woolford, jun., and R. W. Trombitas (Lederle Lab. Div., American Cyanamid Co., Pearl River, N.Y., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1957, **40** (3), 922–926.—Two 10-ml aliquots of the acid extract of the sample are prepared. One is treated (in the order given) with 5 *N* HCl (12 ml), buffer (pH 7·5) (15 ml), $NaHSO_3$ soln. (10 g per 100 ml) (2 ml) and 5 *N* NaOH (3 ml), and heated for 7 min. at 100° to convert the chlortetracycline into anhydrochlortetracycline. The other aliquot (as blank) is treated with buffer (pH 7·5) (15 ml), $NaHSO_3$ soln. (2 ml) and 5 *N* NaOH (3 ml), and heated, first at 100° for 5 min., then with 5 *N* HCl (12 ml) for a further 2 min. *iso*Chlortetracycline, which does not afford the anhydro compound, is thus formed. The difference in extinction at 445 mμ is then measured. The method is accurate, reproducible and rapid. The buffer is a filtered soln. of K_2HPO_4 (178 g) and KH_2PO_4 (22 g) in water (1 litre).
A. A. ELDRIDGE

1014. **The identification of insecticide residues on plant material (fruit and vegetables) and the isolation of pesticides by means of paper chromatography.** R. Müller, G. Ernst and H. Schoch (Canton Lab., Basle, Switzerland). *Mitt. Lebensmitt. Hyg., Bern*, 1957, **48** (3), 152–197.—Methods are described for the extraction, isolation and identification of small quantities (10 to 200 μg) of aldrin, dieldrin, DDT, hexachloro*cyclo*hexane, parathion, chlorthion [O-(3-chloro-4-nitrophenyl) OO-dimethyl phosphorothioate], diazinon and malathion.
H. F. W. KIRKPATRICK

1015. **Analysing new herbicides and insecticides.** K. Gardner (Fisons Pest Control, Ltd., Harston, Cambs). *Chem. Prod.*, 1957, **20** (5), 187–190.—Methods of analysis, and the relevant literature, concerning the analysis of the following materials are reviewed: D.N.C. (2-methyl-4:6-dinitrophenol), C.M.U. (N-*p*-chlorophenyl-N′N′-dimethylurea), S.T.C.A. (sodium trichloroacetate), sodium αα-dichloropropionate, P.C.P. (pentachlorophenol), DDT, BHC, aldrin, dieldrin, schradan, dimefox, methyl demeton, parathion and malathion. (32 references.)
S.C.I. ABSTR.

1016. **Critical points in the Schechter - Hornstein colorimetric method for lindane [γ-hexachlorocyclohexane].** C. W. Gehrke, J. L. Bevirt and M. R. Johnston (Dept. Agric. Chem. and Hort., Coll. of Agric., Univ. of Missouri, Columbia, U.S.A.). *J. Agric. Food Chem.*, 1957, **5** (6), 444–446.—The method was critically examined to improve precision and accuracy, in a study of the residual concn. of lindane in pickles. Difficulty was encountered in obtaining a workable standard curve. The results were erratic, partly because of the use of H_3PO_4 as a lubricant on the ground glass seals of the reaction flasks and the nitrating apparatus. The precision was improved significantly when acetic and dichloroacetic acid lubricants were used; the average deviation for the three acids was 16·1, 3·6 and 5·3%, respectively.
S.C.I. ABSTR.

1017. **Determination of aldrin and isodrin.** J. Prat and A. Colas. *Chim. Anal.*, 1957, **39** (8), 298–300.—This bromimetric method is suitable for the determination of pure aldrin (1:2:3:4:10:10-hexachloro-1:4:4*a*:5:8:8*a*-hexahydro-*exo*-1:4-*endo*-5:8-dimethanonaphthalene) in the technical product, the impurities in the technical material not being brominated under the conditions given. The determination is carried out in a special glass-stoppered flask (illustrated), which has a deep flange surrounding the stopper and is divided into two compartments, upper and lower, by a glass partition extending horizontally about half-way across the flask about one-third of the way from the bottom. The sample (300 to 350 mg) is dissolved in 25 ml of acetic acid and transferred to the lower compartment of the flask and washed in with three further 25-ml portions of acid, followed by 5 ml of conc. HCl. The flask is placed in an inclined position, and 25 ml of 0·1 *N* bromide - bromate is placed in the upper compartment. The flask is then stoppered, the contents are mixed by shaking, and a few ml of water are placed in the flange to seal the stopper against loss of Br vapour. The flask is set aside in the dark for 45 min., and 200 ml of water is then added cautiously, with the stopper only partially lifted, followed by 10 ml of 10% KI soln. After being shaken, the contents of the flask are titrated with 0·1 *N* $Na_2S_2O_3$. Recoveries of pure aldrin ranged from 99·6 to 99·8%, with satisfactory recovery from technical aldrin and from known mixtures. The precision is ± 0·2%. The results for 80 to 100-mg samples of aldrin were satisfactory. When applied to isodrin (1:2:3:4:10:10-hexachloro-1:4:4*a*:5:8:8*a*-hexahydro-*exo*-1:4-*exo*-5:8-dimethanonaphthalene), recoveries of 99·8 and 100% from pure, and of 95·5 and 95·9% from technical, samples were obtained.
R. E. ESSERY

1018. **The purification of charcoal for removing organic colouring in soil extracts before phosphate estimations.** M. H. Bertheux (Univ. Coll. of Ghana, Achimota, Accra). *Chem. & Ind.*, 1957, (35), 1183–1184.—An earlier purification process of boiling with conc. HCl and washing with NaCl reduced the phosphorus content of 0·12 g of charcoal from 0·2 to 0·001 mg. This was too high for charcoal used in decolorising soln. for phosphate estimation. By boiling this pre-treated charcoal with Na_2SO_4 and H_2SO_4, the phosphorus content was reduced from 0·001 to 0·0002 mg. The complete procedure comprises boiling the charcoal with HCl, filtering, reboiling, washing free of Cl^-, boiling with Na_2SO_4 and H_2SO_4, and washing free of SO_4^{2-}. F. J. M.

See also Abstracts 796, 866, 869, 871, 970.

5.—GENERAL TECHNIQUE AND LABORATORY APPARATUS

General

1019. **Micro-pipette for remote handling operations.** K. R. Hyde (Chem. Div., A.E.R.E., Harwell, England). *Chem. & Ind.*, 1957, (35), 1184.—This apparatus, designed for sampling small volumes of materials of high γ-activity, is made from capillary tubing of previously determined bore. One end is blown into a bulb, which fits into a tong head and is provided with an electric heating coil and an air jet. The liquid is drawn into the tube by alternately heating and cooling the bulb, and the length of the thread of liquid is measured by a graduated scale or travelling microscope; the volume is deduced from the calibration data. The accuracy of the sample length, as measured by the graduated scale, is $\pm$ 2%. F. J. M.

1020. **Apparatus for use in quantitative chemical determination processes.** Central Electricity Authority and E. C. Potter. Brit. Pat. 786,272; date appl. 20.10.54.—The apparatus is mainly designed for the addition of small quantities of reagents for the determination of dissolved oxygen in boiler-feed water, steam condensates, etc. It comprises an improved sampling tube and three-way cock which differs from the conventional gas-sampling tube provided with a three-way cock in that the first way is at all times in communication with the interior of the vessel, so that there is no "dead" vol. to be taken into account when the plug is in such a position that the second way communicates with the interior of the vessel through the first way. J. M. Jacobs

1021. **Apparatus for the analysis of mixtures of gases by means of gas absorption percolation.** N. V. de Bataafsche Petroleum Maatschappij [Inventors: A. I. M. Keulemans and A. Kwantes]. Brit. Pat. 784,169; date appl. 22.4.55.—An arrangement for vapour-phase chromatography is described in which the absorption column, a device for separating a component or group of components of the mixture from the carrier gas which emerges from the column, and a detection instrument for measuring a physical property such as thermal conductivity are situated in a single temperature-controlling device. N. E.

1022. **Improvements relating to methods of and apparatus for analysing gases.** H. Feichtinger. Brit. Pat. 785,815; date appl. 19.8.55. Germany, date appl. 21.8.54.—In the analysis of the flue gases of blast-furnaces for the purpose of controlling the operation of the furnace, the mixture of gases to be analysed is forced, step by step, through a series of interconnected chambers, in each of which the mixture is caused to react with an absorbent or other reagent, by means of a conveying liquid. The vol. of the mixture entering or leaving each chamber at a constant pressure is determined by measuring the vol. of conveying liquid required to force the gas mixture past a measuring mark on the capillary tubes connecting the chambers. J. M. Jacobs

1023. **Improvements in or relating to sonic gas analysers.** C. A. Parsons & Co. Ltd. [Inventors: A. E. Martin and D. Mounfield]. Brit. Pat. 783,748; date appl. 30.12.54.—The phase change (dependent on the nature of the gas in one of the sound paths) between voltages derived from two sound-receiving transducers is compared. The phase change is determined by applying one voltage between one grid and the cathode and the other between a second grid and the cathode of a multi-grid valve. Direct current changes in anode currents, as the phase angle between the two voltages varies, is indicated on a meter. J. M. Jacobs

1024. **Improvements in or relating to sonic gas analysers.** Sir Howard Grubb Parsons & Co. Ltd. [Inventors: D. Mounfield and A. E. Martin]. Brit. Pat. 784,146; date appl. 25.10.54 and 9.2.55.—Sonic gas analysers, which depend on the fact that the velocity of sound in a gas varies from one gas to another, are calibrated by a method in which a phase-shift, corresponding to a known concentration of gas, is produced electrically and is arranged to give a deflection of zero to full scale on the indicating meter. J. M. Jacobs

1025. **Improvements in or relating to sonic gas analysers.** C. A. Parsons & Co. Ltd. [Inventors: A. E. Martin and D. Mounfield]. Brit. Pat. 785,000; date appl. 17.1.55.—Changes in the velocity of sonic or ultrasonic vibrations through the gas are measured as a phase change between two a.c. voltages. One or more temp.-sensitive elements, *e.g.*, resistors, inductances or capacitors, are associated with the transmitter circuit and/or the receiving transducer circuit so as to produce a phase change which compensates for changes in the velocity of the vibrations through the gas due to temp. changes in the gas. J. M. Jacobs

1026. **New method of continuous gas analysis.** G. M. Schwab and O. Neuwirth. *Chem.-Ing.-Tech.*, 1957, **29**, 345–347.—The apparatus consists of a number of identical capillary flow-meters connected in series and separated by absorption vessels. Flow rates obtained from pressure-drop readings cannot be compared directly, as they depend on the viscosity of the gaseous mixture, and the Hagen-Poiseuille equation must be applied. H. Sawistowski

1027. **Greaseless gas-measuring receiver for Toepler pump.** R. M. Roberts and J. J. Madison (Shell Development Co., Emeryville, Calif., U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1555.—The apparatus described consists of a gas pipette mounted directly above the pump with a mercury check valve between the pipette and pump to keep the gas in the pipette. The pump, of conventional design, is fitted with 3 gas-tight tungsten electrodes to allow automatic operation. K. A. Proctor

1028. **Apparatus for the preparation, analysis and investigation of plutonium solutions.** D. E. Glanville, D. W. Grant and G. L. Strachan (A.E.R.E., Harwell, England). A.E.R.E. Report C/R 2188, 1957, 24 pp.—The use of the A.E.R.E. Mk. 1A glove box is described and illustrated in connection with operations such as weighing, vacuum filtration, the use of pipettes and burettes, electrolytic oxidation and reduction, ignitions at 1200°, sealing of glass and silica ampoules with a carbon arc, and spectrophotometry at temperatures up to 180°.

G. J. HUNTER

1029. **Micro-hydrogenation sample technique for volatile compounds.** R. M. Engelbrecht (Res. Dept., Lion Oil Div., Monsanto Chemical Co., El Dorado, Ark., U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1556.—The technique described overcomes difficulties such as the excessive time of dissolution with the gelatin capsule method, accurate weighing of milligram quantities, and volatilisation during the removal of air and while the apparatus is reaching equilibrium. K. A. PROCTOR

1030. **Filtration apparatus for organic gravimetric analysis.** A. N. Fletcher (Anal. Chem. Branch, U.S. Naval Ordnance Test Sta., China Lake, Calif., U.S.A.). *Anal. Chem.*, 1957, **29** (9), 1387–1388.—The apparatus described can be used for the suction filtration of such materials as oxidised products from lubricating oils, hydrolysed cellulosic solids and gelatinous ppt. K. A. PROCTOR

1031. **Two-piece centrifuge crucible for handling microchemical precipitates.** C. Feldman and J. Y. Ellenburg (Oak Ridge Nat. Lab., Tenn., U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1557.—The device described allows a micro ppt. to be further processed without changing containers, so as to reduce or eliminate losses and contamination. It consists of a centrifuge tube which can be drained and separated into 2 parts, the lower of which can be used for drying, ignition or dissolution of the ppt.

K. A. PROCTOR

1032. **The "Spinco" ultracentrifuge.** Anon. *Chem. Prod.*, 1957, **20** (5), 208–209.—The apparatus described comprises a means for driving various rotors at constant speeds (up to 70,000 r.p.m.), an optical system of the refractive-index type with viewing screen and automatic camera, high-vacuum pumps for evacuating the rotor chamber, and refrigeration gear for low-temp. operation. The apparatus permits the centrifugal purification of particles of 1 μ down to a few mμ in size and determination of mol. wt. by either the sedimentation-velocity or the equilibrium method.

S.C.I. ABSTR.

1033. **Analytical distillation in miniature columns. Design and testing of Teflon spinning band.** A. G. Nerheim [Res. Dept. Standard Oil Co. (Indiana), Whiting, U.S.A.]. *Anal. Chem.*, 1957, **29** (10), 1546–1548.—The efficiency of spinning band fractionating columns has been increased by using a 4-bladed Teflon band, which incorporates the advantages of low pressure drop, small hold-up, and a large number of plates in a single column.

K. A. PROCTOR

1034. **Improved splash tube for drop counting fraction collectors.** J. S. Baumstark (Dept. of Agric. Chem., Univ. Missouri, Columbia, U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1561.—The tube described overcomes the difficulties of aligning a long chromatographic tube (165 cm) over the open splash tube of a photo-tube unit in an automatic fraction collector. The need for re-evaluation of drop size is also eliminated if a different type of column is used with the same liquid phase.

K. A. PROCTOR

1035. **A form of siphon with variable capacity as a fraction collector for column chromatography.** P. Dupuy and C. Moreau (Lab. de Biol. Vég., Fac. des Sci., Poitiers, France). *Bull. Soc. Chim. France*, 1957, (4), 521.—To the bottom of the cylinder of a 50-ml syringe are fitted a siphon tube and a tube reaching above the height of the siphon bend, open to the atmosphere. A piston with a thin tube through it, having a small funnel on the end, can be adjusted in the cylinder so that siphoning from the cylinder occurs when amounts variable between 7 and 53 ml have been collected.

E. J. H. BIRCH

1036. **A new type of circular paper chromatography.** H. Sulser (Lab. des Eidg. Gesundheitsamtes, Bern, Switzerland). *Mitt. Lebensmitt. Hyg., Bern*, 1957, **48** (2), 117–120.—The substances to be compared are placed on a wedge-shaped constriction of filter-paper produced by making four incisions, with scissors, into the middle of the base of a filter-paper folded into a semi-circle. It is claimed that a high degree of separation is attained as the result of a radial spread of the solutions. P. HAAS

1037. **Paper chromatography in a centrifugal field.** H. J. McDonald, E. W. Bermes, jun., and H. G. Shepherd, jun. (Dept. of Biochem., Loyola Univ., Chicago, Ill., U.S.A.). *Naturwissenschaften*, 1957, **44** (1), 9–10.—A shallow steel dish is used as centrifuge head. This accommodates a circular filter-paper 45 cm in diameter. The soln. under nvestigation is applied as a narrow ring zone close to the centre of the paper. Whilst in motion the developer is added from the tip of a micro-pipette to provide a continuous stream. The pipette is connected to an Erlenmeyer flask which in turn is connected to a gas (helium) cylinder, and the connections are made in such a way that by regulating the gas pressure the flow-rate of the pipette can be finely controlled. E. KAWERAU

1038. **Chromatographic apparatus allowing concentration of constituents.** R. Audran and G. Reutenauer. *Compt. Rend.*, 1957, **245** (2), 168–171.—The principle of the apparatus described is to allow the soln. being chromatographed to ascend a filter-paper cone, the solvent being evaporated near the apex of the cone.

S.C.I. ABSTR.

1039. **Quantitative evaluation of spots on paper chromatograms. Light-flux measurement through negative printed spots.** R. Mykolajewycz (Olin Mathieson Chem. Corp., New Haven, Conn., U.S.A.). *Anal. Chem.*, 1957, **29** (9), 1300–1305.—A negative photocopy of the developed chromatograms is printed. The spots appear more or less white on a black background and are transluscent when illuminated from one side. The intensity of light transmitted through the paper is measured with a photometer and is approximately linear with respect to the quantify of substance in the spot; it is also virtually independent of the form and size of the spot. Application of the method to amino acids gave an average deviation from the mean of 5% for tyrosine; lactobionic acid showed 8% deviation when only one spot was measured.

G. P. COOK

1040. **Improved contact printing frame for ultra-violet light absorbing compounds separated on paper by chromatography or electrophoresis.** D. S. Kinnory and J. Greco (Radioisotope Service, Veterans Admin. Hospital, Hines, Ill., U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1562–1563.—X-ray film with the paper chromatogram above it is held firmly against a convex backing plate during exposure to u.v. D. A. PANTONY

1041. **Constant-rate flow device for electrolyte eluents in column chromatography.** R. K. Main, L. J. Cole, L. M. Bryant and S. K. Morris (Biolog. and Med. Sci. Div., U.S. Naval Radiological Defense Lab., San Francisco, Calif., U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1558–1560.—The device described is capable of delivering a constant fixed vol. (from 3 to 5 ml) of electrolyte per hour for periods of unattended operation up to two weeks.
K. A. PROCTOR

1042. **Detectors for vapour-phase chromatography.** J. R. M. *Chem. Prod.*, 1957, **20** (5), 206–207.—The following types of detector are described—katharometer, volumetric gas collector, titration cell, microflame - thermocouple combination, and a simplified Martin gas density balance.
S.C.I. ABSTR.

1043. **Transfer cell for gas chromatography.** D. S. Russell and M. E. Bednas (Nat. Res. Council, Ottawa, Canada). *Anal. Chem.*, 1957, **29** (10), 1562.—Details and manipulation of a simple transfer cell for quant. transfer of extremely small (10^{-6} mole, minimum) gas samples are given.
D. A. PANTONY

1044. **Thermal conductivity behaviour. Importance in quantitative gas chromatography.** D. M. Rosie and R. L. Grob (Products Res. Div., Esso Res. and Engng Co., Linder, N.J., U.S.A.). *Anal. Chem.*, 1957, **29** (9), 1263–1264.—The thermal conductivity behaviour of a variety of hydrocarbons has been studied with 2-metre columns of celite impregnated with dinonyl phthalate. It was shown that the difference in cell response for similar hydrocarbons may be significantly large. By making corrections for each compound, precise quant. results can be obtained. K. A. PROCTOR

1045. **Thermal conductivity cell for gas chromatography.** S. A. Ryce, P. Kebarle and W. A. Bryce (Dept. of Chem., Univ. of British Columbia, Vancouver, Canada). *Anal. Chem.*, 1957, **29** (9), 1386–1387.—The inexpensive cell described is made of brass and contains filaments which consist of helices of 0·3 cm in diam. constructed from 14 cm of 0·005-cm platinum wire. The reference and detecting channels are symmetrical and the filaments are located in the middle of the gas stream. The minimum amount of material detectable is approx. 10^{-8} mole, and the noise level is insignificant at max. sensitivity. K. A. PROCTOR

1046. **Ion-exchange resin membranes.** E. B. Trostyanskaya, I. P. Losev and A. S. Tevlina (D.I. Mendeleev Moscow Chem.-Tech. Inst.). *Zhur. Anal. Khim.*, 1957, **12** (2), 214–219.—The advantages of the use of ion-exchange resin films and membranes made from the usual ion-exchange resins are discussed. G. S. SMITH

1047. **Theory of gradient elution through ion exchangers.** H. Schwab, W. Rieman, III, and P. A. Vaughan (Wright Chem. Lab., Rutgers Univ., New Brunswick, N.J., U.S.A.). *Anal. Chem.*, 1957, **29** (9), 1357–1361.—Equations for predicting the position of the peak in the graph of a gradient elution have been derived, and tested experimentally. G. S. ROBERTS

1048. **Determination of the viscosity of bitumen.** J. Zemplén and P. Szabó (Inst. Exp. Phys. Building and Traffic, Univ., Budapest). *Kolloidzschr.*, 1957, **153** (1), 36–38.—Viscosities up to 5×10^7 poises can be measured directly in the type of viscometer described. The bitumen is placed in a brass cylinder and compressed by a piston loaded by known weight. The bitumen is forced out through a capillary 17 mm long and 1 mm in diam., and the weight extruded in a given time is measured. Poiseuille's equation is used. The temp. can be controlled and measured. The normal pressure used is 20 atm. It is found that log η at first decreases with increasing pressure, and then becomes constant. There is an approx. linear relation between log η and $1/T$ at constant pressure.
A. B. DENSHAM

Optical

1049. **Extrusion of cylindrical specimens for X-ray powder diffraction analysis.** L. J. E. Hofer, A. Damick, A. F. Headrick, F. Fauth, E. H. Bean and P. L. Golden (Coal-to-Oil Res. Branch, Bureau of Mines, Bruceton, Pa., U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1563–1564.—Certain aspects of the manual extrusion procedure of Jellinek and Fankuchen (*Ind. Eng. Chem.*, 1945, **37**, 158) are described. K. A. PROCTOR

1050. **Device for varying burner height in Beckman flame photometer.** I. May, H. Kramer and E. L. Curtis (U.S. Geol. Survey, Washington, D.C., U.S.A.). *Anal. Chem.*, 1957, **29** (9), 1388.—An attachment is described for reproducibly raising or lowering the atomiser-burner of a Beckman flame photometer. In this way different parts of the flame can be brought into focus on the slit without changing the mirror adjustment. K. A. PROCTOR

1051. **Simple infra-red and ultra-violet photometers in analytical chemistry.** A. Berton. *Chim. Anal.*, 1957, **39** (8), 288–297.—Descriptions and diagrams are given of several types of flame photometers and analysers, and an attachment for the direct photographic registration of the movements of the galvanometer spot without the necessity for chemical development. Numerous examples of the application of these instruments are given, including the determination of vitamins, hormones and antibiotics, of light water in heavy water, of moisture in organic liquids, of CO_2 and toxic vapours in air, and of organic substances containing strongly absorbing groups such as OH, NH, CO, CN. Certain reactions can be followed, such as the oxidation of fats, and separations, as in gas- and liquid-phase chromatography. R. E. ESSERY

1052. **Internally standardised general spectrographic method.** A. J. Frisque [Standard Oil Co. (Indiana), Whiting, U.S.A.]. *Anal. Chem.*, 1957, **29** (9), 1277–1279.—A large number of elements in a wide variety of matrices can be determined by this method with errors of $< 25\%$. A 1:3 mixture of germanium dioxide and graphite is used as internal standard, buffer and diluent. The use of this single matrix and of an improved powder-handling technique minimises the analysis time.
K. A. PROCTOR

1053. **Use of the didymium filter for wavelength calibration in spectrophotometry.** R. S. Melville (Veterans Admin. Hospital, Iowa City, U.S.A.). *Proc. Iowa Acad. Sci.*, 1956, **63**, 419–422.—The use of this filter, composed of neodymium dispersed in glass, for wavelength calibration of clinical spectrophotometers is described and discussed. A method for ensuring correct alignment of the instrument involves verifying the calibration at 610 mμ and then re-setting the wavelength of the instrument to 600 mμ; the second reading should then be considerably lower than the first.

O. M. WHITTON

1054. **The spectrographic analysis of metals.** J. O. Lay. *Metallurgia, Manchr*, 1957, **55**, 307–308.—The operation and useful range of the Steeloscope and the medium and large quartz spectrographs are indicated briefly. With the Steeloscope the two electrodes have the same composition and the spectrum is examined visually with an eyepiece marked and adjusted so that the characteristic line of an element can be brought to the centre of the visible part of the spectrum image for identification and measurement of intensity (based on standard samples). The wavelength spectrometer is a similar instrument for use on non-ferrous metals, and is mainly of value for detecting appreciable differences of composition. The large quartz spectrograph should be used for steel and related alloys, ores and slags. A compact photo-electric spectrograph giving high dispersion, with direct comparison of sample and standard, is now available for routine industrial analysis (*cf.* Tyas, *Anal. Abstr.*, 1957, **4**, 752).

W. J. BAKER

1055. **A projection spectrum comparator.** C. F. Smith (1 Wood End Close, Farnham Common, Bucks., England). *J. Sci. Instrum.*, 1957, **34** (8), 324–326.—A spectrum comparator is described, in which the images of the spectrum plate being examined and a standard spectrum plate are simultaneously projected on a translucent screen by an optical system giving a magnification factor of 20. Shutters are provided for masking any unwanted portions of the images, and a micrometer adjustment for the plate carriages serves both to bring the spectrum lines into coincidence, and, in conjunction with a graticule on the screen, to measure the distance between lines; readings can be taken to 0·0001 in. Full adjustment to the plate mounts is provided, and the adjustments to the optical system include neutral tint wedges for equalising lamp brightness or background level. A photographic attachment is available, with 5 in. × 4 in. plates or films. An additional comparison facility is given by fitting a semi-aluminised mirror, which can be placed so that the projected image is viewed superimposed over a spectrum chart, mounted beneath the mirror.

J. F. P. H. GREENE

1056. **A "split-second" spectrophotometer for the registration of rapid changes in absorption.** D. Lübbers and W. Niesel (Physiol. Inst. der Univ., Kiel, Germany). *Naturwissenschaften*, 1957, **44** (3), 60.—The instrument is an electronic spectrophotometer which can record an extinction curve over a wide spectral range in 1/120th second; the results can be evaluated on a quant. basis. The instrument will be used for a study of the rapid changes that take place in the respiratory enzymes of biological systems. A short description and sketch of the instrument are given and a note preceding the paper discusses the physical laws governing the construction.

E. KAWERAU

1057. **Paper-strip holder for absorption spectroscopy.** R. M. Horowitz and L. F. Atkinson (Fruit and Veg. Chem. Lab., U.S. Dept. of Agric., Pasadena, Calif., U.S.A.). *Anal. Chem.*, 1957, **29** (9), 1385.—The holder described is designed specifically for use in a Beckman Model DU spectrophotometer. It can be used to measure the entire spectrum of a particular spot on a chromatogram, but it is not suitable for scanning an entire chromatogram at one wavelength.

K. A. PROCTOR

1058. **Two new histophotometers for study of absorption curves.** M. Vialli and L. Zanotti (Ist. Anatomia Comp., Univ. Pavia). *Ric. Sci.*, 1957, **27** (3), 814–824.—The histophotometers described facilitate the interpretation of absorption curves and can also be used for u.v. studies. One model has the blank optic axis outside the optical system of the microscope. The other has two diaphragms which can be centred independently from each other and are located above the microscope, one for the object, the other for the blank. By means of a quartz lens the rays from the two diaphragms are focused alternately on the same area of the photocathode of a photomultiplier.

L. KRUSZYNSKA

1059. **Infra-red spectroscopy from 5 to 200 microns with a small grating spectrometer.** R. C. Lord and T. K. McCubbin, jun. (Spectroscopy Lab., M.I.T., U.S.A.). *J. Opt. Soc. Amer.*, 1957, **47** (8), 689–697.—Modifications made to standard spectrometers, so that they operate with gratings, photoelectric detectors, and the Walsh double-pass system, are described. Two instruments are described, one for use in the 5 to 70-μ region, and the other from 50 to 200 μ. Very complete information on the modifications is given, with diagrams and drawings of some parts. Tables and curves for several calibration and test runs are also given. Between 5 and 50 μ, a resolution of 0·03 μ is claimed, and, for the range above 50 μ, a resolution varying from 0·05 to 0·1 μ.

J. F. P. H. GREENE

1060. **Reduced-scale auxiliary recording of infra-red spectra.** F. Stitt and G. F. Bailey (Agric. Res. Service, U.S. Dept of Agric., Albany, Calif., U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1557.—A simple, convenient auxiliary recording system for producing reduced-scale i.r. curves simultaneously with the standard instrument record is described for use with the Beckman Model IR–3 spectrophotometer. The system is adaptable for use with other instruments and is built from readily available components.

K. A. PROCTOR

1061. **2:2-Dimethoxypropane as a drying agent for preparation of infra-red samples.** D. S. Erley (Dow Chemical Co., Midland, Mich., U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1564.—A non-volatile sample which is slightly acidic or stable in a slightly acid environment (pH < 6) can be easily dried for i.r. analysis by adding excess of 2:2-dimethoxypropane. The products of the reaction with water (acetone and methanol), as well as any excess reagent, usually evaporate on standing or on grinding with liquid paraffin.

K. A. PROCTOR

1062. **Quantitative infra-red vapour spectra.** R. A. Friedel and J. A. Queiser (Central Exp. Sta., U.S. Bureau of Mines, Bruceton, Pa., U.S.A.). *Anal. Chem.*, 1957, **29** (9), 1362–1366.—The quant. introduction of volatile components into an evacuated i.r. gas cell can be achieved by discharging liquids

from a self-filling micro-pipette through a sintered disc. The method can be used for single components or mixtures and is applicable to aq. soln. Among advantages of the method are the production of fine structure and the elimination of solvent. Disadvantages include pressure-broadening and sorption of some vapours in the cell.

K. A. PROCTOR

1063. **Compton scattering interference in fluorescent X-ray spectroscopy.** E. J. Brooks and L. S. Birks (U.S. Naval Res. Lab., Washington, D.C., U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1556.—In fluorescent X-ray spectroscopy, interference may be caused not only at the primary X-ray wavelengths but also at wavelengths corresponding to the Compton scattering associated with the primary wavelengths. This is particularly so when the matrix contains mostly light elements, such as in common plastics and hydrocarbon liquids.

K. A. PROCTOR

1064. **Organometallic precipitates and briquetting for X-ray spectrography.** J. E. Fagel, jun., E. W. Balis and L. B. Bronk (Res. Lab., General Electric Co., Schenectady, N.Y., U.S.A.). *Anal. Chem.*, 1957, **29** (9), 1287–1289.—Briquetted organometallic ppt. are the basis of a general method of analysis by X-ray emission spectrography. Briquetting can also be used for the prep. of samples from homogeneous solids such as metallic powders, trimmings or chips. The density of the briquettes must be controlled within 5% to avoid appreciable errors in the measured X-ray intensities.

K. A. PROCTOR

1065. **Fluorimetric uranium analyser.** J. T. Byrne (Rocky Flats Plant, Dow Chemical Co., Denver, Colo., U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1408–1412.—The construction and electrical circuits of a fluorimeter are described. Fluorescence from a uranium - sodium fluoride phosphor is measured by means of a photomultiplier tube, and compensation for variations of voltage and lamp intensity is made by means of a second tube which monitors the incident u.v. light. The sensitivity is given as 2 to 4 $\times$ 10^{-9} g of U. The effects of temperature and fatigue are discussed. D. A. PANTONY

1066. **Refractometer.** Carl Zeiss-Stiftung. Brit. Pat. 785,563; date appl. 27.7.55; Germany, date appl. 10.8.54.—The refractive index of liquids is measured by the determination of the critical angle of total reflection. By measuring the reflected light, the effect of absorption of light by the sample under examination is eliminated. Structural details are given. J. M. JACOBS

1067. **X-ray shadow microscopy as an aid for the analyst.** Shigeto Yamaguchi [Sci. Res. Inst., Ltd., 31 Kamifuji (Hongo), Tokyo, Japan]. *Anal. Chem.*, 1957, **29** (9), 1387.—X-ray shadow microscopy can be used for the preliminary detection of such elements as Th and U in powdered samples.

K. A. PROCTOR

Thermal

1068. **Apparatus for determining the gas content of metals.** Z. M. Turovtseva, N. F. Litvinova, G. V. Mikhallova, A. S. Noskov and R. Sh. Khalitov (V.I. Vernadskiĭ Inst. of Geochem.). *Zhur. Anal. Khim.*, 1957, **12** (2), 208–213.—Apparatus for vacuum fusion with means for continuous collection of the liberated gases and their analysis is described.

G. S. SMITH

1069. **Simplification of a semi-micro zone melting apparatus.** R. Handley and E. F. G. Herington (Chem. Res. Lab., Teddington, England). *Chem. & Ind.*, 1957, (35), 1184.—The combination of parabolic and spherical mirrors in the earlier apparatus is replaced by a single 7-in. aluminium ellipsoidal reflector of 2 in. minor and 14 in. major foci. The projection lamp is placed at the focus nearer the mirror and the specimen at the other. The image is larger than with the previous apparatus, but the width of the molten zone may be reduced by placing a tin-foil screen in front of the i.r. filter glass. F. J. M.

1070. **Micro-ebulliometer for determination of molecular weight.** M. Dimbat and F. H. Stross (Shell Development Co., Emeryville, Calif., U.S.A.). *Anal. Chem.*, 1957, **29** (10), 1517–1520.—The apparatus described can be used for the convenient and precise determination of mol. wt. on as little as 10 mg of sample. High sensitivity is attained by the use of small solvent vol. (5 ml) and the use of thermistors for measuring temp. differences as small as 6 $\times$ 10^{-5}°. The percentage errors in measuring once each the mol. wt. of six pure compounds in benzene were +0·7, 0·0, −0·6, −0·4, 0·0 and +0·4.

K. A. PROCTOR

1071. **Use of thermogravimetry in analytical chemistry. I. A thermobalance of simplified design.** P. N. Palei, I. G. Sentyurin and I. S. Sklyarenko (V.I. Vernadskiĭ Inst. of Geochem. and Anal. Chem., Acad. Sci., USSR, Moscow). *Zhur. Anal. Khim.*, 1957, **12** (3), 318–323.—A simple thermobalance is described. With an original sample wt. of 30 to 50 mg, wt. changes with temp. of up to 20 mg can be continuously recorded. Scale divisions are in 0·1 mg, but readings can be made to 0·01 mg. G. S. SMITH

1072. **Investigations with the help of thermogravimetry.** L. Erdey (Tech. Univ., Budapest, Hungary). *Period. Polytech.*, 1957, **1** (1), 35–50.—The advantages to be gained by correlating the results obtained by the methods of differential thermoanalysis, thermogravimetry and derivative thermogravimetry are reviewed. The investigation of the composition of several minerals, industrial raw materials, solid and liquid fuels and analytical ppt. by the three methods together is discussed and the conclusions to be drawn are stressed. In the course of investigation of proton-free acid - base reactions some regularity in the magnitude and sequence of basic strengths in Lewis acids and bases was observed. E. G. CUMMINS

1073. **Variation of peak temperature with heating rate in differential thermal analysis.** H. E. Kissinger. *J. Res. Nat. Bur. Stand.*, 1956, **57** (4), 217–221.—In differential thermal analysis the temp. at which the max. deflection is observed varies with heating rate in certain types of reactions. An expression has been derived relating this variation with the kinetics of the reaction. Measurements of the variation of peak temp. with heating rate have been made for several minerals of the kaolin group, and the values of the kinetic constants have been determined and compared with corresponding values obtained by conventional isothermal techniques. N. E.

1074. **Measurement of the proportion of constituents in mixtures.** P. Gilli. Brit. Pat. 784,218; date appl. 20.1.54; Austria, date appl. 21.1.53,

18.6.53 and 22.6.53.—The proportions of constituents in a flowing medium are indicated mechanically by transferring heat to or from the medium. The medium is caused to flow through a measuring pipe at a constant rate at so high a speed that the changes in length of the measuring pipe in relation to the length of a comparison pipe (these relative changes in length being caused to give the mechanical indication) are due to the heat transfer to or from the medium and to the specific heat of the medium, and are not due solely to the heat conductivity of the medium. The method is particularly suitable for the measurement of the CO_2 content of flue gases. J. M. JACOBS

Electrical

1075. **Zone electrophoresis in columns.** J. Porath. *Chem. Weekbl.*, 1957, **53** (40), 533–536 (in English).—Proteins, nucleic acid and other polyelectrolytes can be fractionated by electrophoresis in vertical cellulose columns. Annular columns cooled internally and externally are described. In the largest, the electrolyte is circulated between the electrode compartments and a cooling chamber. The smallest column described is suitable for samples of 30 to 40 g in 50 to 250 ml of soln. and the largest for samples of 140 g in 1 to 2 litres of soln. Aminopeptidase has been prepared in this way sufficiently pure for end-group studies of proteins. Crystalline enolase has been separated into three fractions. Other examples are cited. G. BURGER

1076. **Apparatus for separation of substances by paper electrophoresis.** T. Poch (Chem. Tech. Res. Inst., Meinsberg). *Chem. Tech., Berlin*, 1957, **9** (5), 274–279.—The development of paper electrophoresis is reviewed, and various types of apparatus are described. C. A. CURTIS

1077. **High-tension electrophoresis and safety measures.** F. Oehme and K. Selkmann (Dtsch. Akad. der Wissenschaften, Berlin). *Chem. Tech., Berlin*, 1957, **9** (4), 221.—A readily made instrument is described with which electrophoresis can be carried out at high tension, up to 10 kV and field intensities of 50 to 100 V per cm. Safety measures are described and a circuit diagram and details of the safety contacts and signals are given. By working under high tension a shorter working time and better separation of zones are claimed. C. A. CURTIS

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Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use of an abbreviation or symbol the word is printed in full.

alternating current	a.c.
ampere	amp.
Ångström unit	Å
anhydrous	anhyd.
approximate, -ly	approx.
aqueous	aq.
atmospher-e, -ic	atm.
boiling-point	b.p.
British thermal unit	B.Th.U.
calorie (large)	kg-cal.
calorie (small)	g-cal.
centimetre	cm
coefficient	coeff.
concentrated	conc.
concentration	concn.
constant	const.
corrected	(corr.)
crystalline / crystallised	cryst.
cubic	cu.
current density	c.d.
cycles per second	c/s
density	ρ
density, relative	d or wt. per ml
dilute	dil.
direct current	d.c.
distilled	dist.
ethylenediaminetetra-acetic acid	EDTA
electromotive force	e.m.f.
equivalent	equiv.
gram	g
gram-molecule	mole
half-wave potential	$E_{\frac{1}{2}}$
hour	hr.
hydrogen ion exponent	pH
infra-red	i.r.
insoluble	insol.
international unit	i.u.
kilogram	kg
kilovolt	kV
kilowatt	kW
liquid	liq.
maxim-um, -a	max.
melting-point	m.p.
microgram	μg (not γ)
microlitre	μl
micromole	μmole
micron	μ
milliampere	mA
milli-equivalent	milli-equiv.
milligram	mg
millilitre	ml
millimetre	mm
millimicrogram	mμg
millimolar	mM
millivolt	mV
minute (time)	min
molar (concentration)	M
molecul-e, -ar	mol.
normal (concentration)	N
optical rotation	α_λ^t
ounce	oz
parts per million	p.p.m.
per cent.	%
per cent. (vol. in vol.)	%(v/v)
per cent. (wt. in vol.)	%(w/v)
per cent. (wt. in wt.)	%(w/w)
potential difference	p.d.
precipitate (as a noun)	ppt.
precipitated	pptd.
precipitating	pptg.
precipitation	pptn.
preparation	prep.
qualitative, -ly	qual.
quantitative, -ly	quant.
recrystallised	recryst.
refractive index	n_λ^t
relative band speed	R_F
relative humidity	r.h.
revolutions per minute	r.p.m.
saponification value	sap. val.
saturated calomel electrode	S.C.E.
second (time)	sec.
soluble	sol.
solution	soln.
specific gravity	sp. gr.
specific rotation	$[\alpha]_\lambda^t$
square centimetre	sq. cm
standard temp. and pressure	s.t.p.
temperature	temp.
ultra-violet	u.v.
vapour density	v.d.
vapour pressure	v.p.
volt	V
volume	vol.
watt	W
wavelength	λ
weight	wt.

In addition, the following symbols may be used in conjunction with numerical values or in mathematical expressions—

greater than	$>$	less than	$<$
not greater than	$\ngtr$	not less than	$\nless$
is proportional to	$\propto$	of the order of, approximately	$\simeq$

The principal Pharmacopoeias are denoted by B.P., U.S.P. or D.A.B., together with the identifying roman numeral or year.

Valency states are represented by a superscript roman numeral, *e.g.*, Fe^{II}, Mo^{V}. Substances in the ionic state are represented by Na^+, Fe^{2+}, Fe^{3+}, etc., for cations and by Cl^-, SO_4^{2-}, PO_4^{3-}, etc., for anions.

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aqueous	aq.
atmospher-e, -ic	atm.
boiling-point	b.p.
British thermal unit	B.Th.U.
calorie (large)	kg-cal.
calorie (small)	g-cal.
centimetre	cm
coefficient	coeff.
concentrated	conc.
concentration	concn.
constant	const.
corrected	(corr.)
crystalline / crystallised	cryst.
cubic	cu.
current density	c.d.
cycles per second	c/s
density	ρ
density, relative	d or wt. per ml
dilute	dil.
direct current	d.c.
distilled	dist.
ethylenediaminetetra-acetic acid	EDTA
electromotive force	e.m.f.
equivalent	equiv.
gram	g
gram-molecule	mole
half-wave potential	$E_{\frac{1}{2}}$
hour	hr.
hydrogen ion exponent	pH
infra-red	i.r.
insoluble	insol.
international unit	i.u.
kilogram	kg
kilovolt	kV
kilowatt	kW
liquid	liq.
maxim-um, -a	max.
melting-point	m.p.
microgram	μg (not γ)
microlitre	μl
micromole	μmole
micron	μ
milliampere	mA
milli-equivalent	milli-equiv.
milligram	mg
millilitre	ml
millimetre	mm
millimicrogram	mμg
millimolar	mM
millivolt	mV
minute (time)	min
molar (concentration)	M
molecul-e, -ar	mol.
normal (concentration)	N
optical rotation	α_λ^t
ounce	oz
parts per million	p.p.m.
per cent.	%
per cent. (vol. in vol.)	%(v/v)
per cent. (wt. in vol.)	%(w/v)
per cent. (wt. in wt.)	%(w/w)
potential difference	p.d.
precipitate (as a noun)	ppt.
precipitated	pptd.
precipitating	pptg.
precipitation	pptn.
preparation	prep.
qualitative, -ly	qual.
quantitative, -ly	quant.
recrystallised	recryst.
refractive index	n_λ^t
relative band speed	R_F
relative humidity	r.h.
revolutions per minute	r.p.m.
saponification value	sap. val.
saturated calomel electrode	S.C.E.
second (time)	sec.
soluble	sol.
solution	soln.
specific gravity	sp. gr.
specific rotation	$[\alpha]_\lambda^t$
square centimetre	sq. cm
standard temp. and pressure	s.t.p.
temperature	temp.
ultra-violet	u.v.
vapour density	v.d.
vapour pressure	v.p.
volt	V
volume	vol.
watt	W
wavelength	λ
weight	wt.

In addition, the following symbols may be used in conjunction with numerical values or in mathematical expressions—

greater than	$>$	less than	$<$
not greater than	$\ngtr$	not less than	$\nless$
is proportional to	$\propto$	of the order of, approximately	$\simeq$

The principal Pharmacopoeias are denoted by B.P., U.S.P. or D.A.B., together with the identifying roman numeral or year.

Valency states are represented by a superscript roman numeral, *e.g.*, Fe^{II}, Mo^{V}. Substances in the ionic state are represented by Na^+, Fe^{2+}, Fe^{3+}, etc., for cations and by Cl^-, SO_4^{2-}, PO_4^{3-}, etc., for anions.

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ANALYTICAL ABSTRACTS

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